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Recovery of Biomass Fly Ash and HDPE in Innovative Synthetic Lightweight Aggregates for Sustainable Geotechnical Applications

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Abstract: Sustainable development principles aim to re-utilize wastes to reduce their impact on the environment. In this context, the present contribution shows preliminary results on the preparation of innovative synthetic lightweight aggregates, starting from biomass-derived fly ash and high-density polyethylene (HDPE), to be used in geotechnical applications. The present work focuses on the manufacturing process of aggregate blends (including the selection of the right proportions of the two components) as well as on the relative determination of (i) physical–chemical properties (i.e., chemical composition, morphological analysis, mineral leachability, water absorption, specific gravity, grain size distribution); (ii) permeability features and (iii) mechanical properties (one-dimensional compression and shear strength behavior). The results, gathered from the new synthetic lightweight aggregates and compared with the corresponding ones obtained in a previous study conducted on natural and synthetic lightweight aggregates, appear promising for a potential utilization in geotechnical engineering.

Keywords: lightweight aggregates; recycling materials; HDPE; biomass fly ash; chemical and physical analyses; geotechnical characterization

1. Introduction

A circular economy is considered a viable and efficient way to pursue a sustainable development. Even if a completely circular economy is not possible, reducing the use of raw materials with finite reserves and limiting discharges into the environment should be prioritized. In civil engineering applications, the use of construction materials based on industrial by-products and recycled waste, with the ability to prevent the exhaustion of natural resources, is surely an example [1,2]. In this view, the biomass valorization [3,4], such as its use as a raw material for the production of energy, transport fuels, chemicals and others, is of particular interest. Indeed, biomass incineration for energy production presents important economic advantages since it enables decreasing the cost of energy and, at the same time, it has considerable environmental benefits due to the elimination of waste. However, on the other side, it has the disadvantage of generating large amounts of fly and bottom ashes that are environmental pollutants. At present, most of these wastes are disposed in lined landfills, but, with the increasing difficulties in the acquisition and development of new waste treatment sites, the disposal of the fly ash, generated from biomass combustion (BFA), represents a serious problem for the energy production companies as well as for the municipalities. Consequently, alternative

solutions to the disposal are required. Recent studies have demonstrated that BFA can be effectively recycled for construction products such as raw materials for ceramic, cement and concrete additives, road stabilization binder, etc. [5–9]. Nevertheless, commercial applications of the biomass ash are, at present, very limited if compared to fly (and bottom) ash derived from other sources, such as coal based thermal power stations (CFA). In fact, in this regard, a critical issue in fly ash utilization is represented by its chemical composition that can be very different if derived from biomass or coal. Typically, the composition of the fly ash from pure biomass depends on different factors, such as the type of biomass, growing area and climate conditions, type of soil, incineration parameters, etc., and therefore it generally contains more alkali derivatives (Na and K) and less alumina (Al_2O_3) than coal fly ash [5]. From a general point of view, there are mainly two classes of fly ash which are used for recycling in civil engineering applications, namely class C and class F ash [10,11]. The main difference between these two classes is in the content of pozzolanic elements (namely silica, alumina, calcium, and iron oxide) that is higher in class C than in class F. According to the above features, the class C ash possesses self-cementing properties, i.e., in presence of water, pozzolanic compounds such as calcium silicate/aluminate hydrate (CSH/CASH) can fill the capillary pores of the ash and bind ash particles together. As a result, class C ash hardens and gets stronger over time, while class F requires the addition of traditional stabilizers, like lime, cement or similar, to effectively enhance the pozzolanic reactions and produce binding compounds. However, it should be taken into account that costs of lime and cement are high and their manufacturing process can result in a high volume of CO_2 emission, which is harmful to the environment [12]. The reuse of high volumes of class F fly ash with low self-cementing properties in civil engineering applications, requires the development of alternative binding agents/stabilizers able to replace the traditional ones.

Recently the reuse of fly ash (or other similar by-products) opportunely blended with plastic wastes, has received increasing attention in the civil engineering practice as an environmentally friendly and cost effective binding/coating agent to produce stabilized/inertized pellets. In fact, due to the increase in the global production of plastics over the years, the generation and disposal of large volumes of plastic waste has become an acute environmental problem, which requires a rapid solution.

This work aims at investigating the feasibility of reusing biomass fly ash in mixture with recycled plastic to produce synthetic lightweight aggregates (SLAs) for geotechnical applications. Among the remarkable advantages, this process would permit the recycling of two different wastes at the same time.

Compared to aggregates based on coal fly ash, where significant research has already taken place, no comprehensive work has been dedicated to aggregates made of Class-F biomass fly ash and HDPE. Therefore, further research is needed.

In this context, the new lightweight synthetic aggregates may be considered a suitable alternative to the commonly used natural lightweight aggregates (NLA), generally manufactured from pumice, volcanic cinders, clay and siliceous rocks. Indeed, in the last years, thanks to their good properties (low density, high strength and low compressibility, enhanced waves attenuation, high permeability, durability, etc.), the over use of NLA, for lightweight concrete production brought about by the growing infrastructural demand, has become a serious issue. This entrains a continuous rise of costs related to their extraction and transportation. When SLAs are produced using industrial by-products like fly ash [13], they could display a lot of advantages with respect to NLA.

At the same time, the potential release into the environment of heavy metals (HMs) and potential toxic metals (PTMs) today represents one of the most pressing problems, when fly ashes are used as the main or co-component of new secondary raw materials [14]. In the present paper, the authors have focused on the leaching of Cu^{2+}, Ni^{2+}, Zn^{2+}, Mn^{2+} and Fe^{2+} metal ions under neutral conditions (UNI EN 12457-2 method [15]). Copper, nickel and zinc are essential elements for life and are micronutrients in trace amounts [16] but their uncontrolled and untreated discharge is toxic to ecosystems [17,18], with toxicity and dangerous effects for humans causing several diseases and serious health problems [19–24].

To the best of our knowledge, few literature studies reported on the synthesis of new SLAs, most of them focusing on the coal-derived fly ash. In particular, a study concerning the feasibility of SLAs composed by a class F of coal fly ash and high density polyethylene (HDPE) plastic waste, was originally conducted by several authors [25]. The two components were compounded, with two different fly ash/HDPE weight ratios, through a blend-extrusion process and finally granulated to form aggregates. Laboratory tests performed on resulting aggregates revealed a pronounced shear strength (comparable or even higher than that exhibited by NLA) and one dimensional compressibility which decreased (i.e., the material become stiffer) with increasing the fly ash/HDPE ratio. Notably, for the higher ratio (80/20) considered in the tests, the stiffness was very similar to that exhibited by normal light weight aggregates consisting of "commercially available expanded clay/shale/slate, developed via heating in a rotary kiln" in a wide range of pressures (up to approximately 2000 kPa). Further results concerning the above-mentioned coal based fly ash-plastic waste aggregates have been reported by some authors [26–28]. The dominant properties requested to a synthetic lightweight aggregate depend on the applications. Restricting the analysis to the geotechnical field, which is the purpose of this paper, the most common applications include: (a) construction of lightweight embankments on soft soils to reduce settlements induced by the own weight of the embankment; (b) placement of lightweight backfill material behind the earth retaining walls to reduce both static and seismic earth pressures; (c) re-profiling natural or artificial slopes with lightweight materials to increase their stability; (d) to install, in soil near existing buildings, protective trenches filled with high damping material to attenuate waves propagation induced by traffic and/or industrial activities; (e) to construct draining trenches using highly permeable materials; (f) to use SLAs as fillers in road pavement systems and (g) to fill underground cavities by low cost and easy to manage materials. The above-mentioned applications cover a wide range of properties, including (i) the size of granules; (ii) specific gravity; (iii) water absorption, (iv) strength and compressibility under both static and cyclic loadings, (v) creep deformations, (vi) cyclic waves damping, (vii) compaction features and (viii) permeability.

In this paper, laboratory tests, specifically addressed to the determination of the aforementioned properties, were used to characterize SLAs from a physical, chemical and geotechnical point of view, in order to decide the most appropriate uses for it. However, an absolute primary role was also plaid by tests able to verify the compliance of the resulting compounds with the requirements concerning environmental pollution, chemical stability and durability with time.

2. Materials and Methods for SLAs Manufacturing and Characterization

For the production of the innovative granular aggregates, two precursor materials, namely biomass fly ash and plastics, were used. In particular, ashes, which appeared as a heterogeneous dark grey powder, came from the combustion of biomasses of a local waste treatment plant, (Ecopiana Company srl). The used plastic was instead a high density polyethylene (HDPE) of commercial type.

A number of chemical–physical characterization tests were used to study both precursor materials, ashes and plastics, as well as the final aggregates. All tests were performed in accordance with the standardized ASTM (American Society for Testing and Materials) procedures.

SEM-EDX analyses were performed on a Phenom Pro-X scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectrometer (EDX) in order to investigate the morphology of both ashes and aggregates. The EDX analysis was used to evaluate the ashes chemical composition. X-ray diffraction (XRD) was performed to study the ashes crystalline structures using a Bruker D2 Phaser (Cu Kα radiation at 30 kV and 20 mA). The diffraction angles 2θ were varied between 10° and 80° in steps of 0.02° and a count time of 5 s per step. The peak attribution was performed on the basis of the JCPDS (Joint Committee on Powder Diffraction Standards) database of reference compounds. The thermal stability of the samples was evaluated by temperature-programmed TGA/DSC (Thermogravimetric Analysis and Differential Scanning Calorimetry) experiments with a Netzsch instrument. The temperature-programmed experiments were carried out in air in the range 25–800 °C and a heating rate of 10 °C/min. Heavy metals concentration was determined using the

ICP-OES instrument (Perkin–Elmer Optima 800, Waltham, MA, USA). The pH value was measured using combined glass electrodes (inoLab pH/Cond 720, WTW, Weilheim, Germany).

Grain-size distribution analyses were performed on representative samples of both fly ash and aggregates for determining their grading features. The specific gravity G_S of the used biomass fly ash and the aggregate granules were determined by performing measurements with pycnometer and hydrostatic balance, respectively.

2.1. Physical and Chemical Properties of the Biomass Fly Ash

Conventional sieve analysis and aerometry were performed on a representative sample of fly ash, in accordance with the standardized procedures ASTM D421 [29] and ASTM D422 [30], respectively, to determine grading characteristics. Figure 1 shows the particle size distribution curve of the biomass fly ash; it is evident that the predominant fractions are silt and sand with percentages equal to about 59.6% and 32.6%, respectively. Given the practically non-plastic nature of the fines, it was not possible to determine Atterberg limits and consistency indices. The specific gravity G_S of the biomass fly ash was 2.69. Standard Proctor compaction tests [31] were also performed, and an optimum water content value of 20.5% and a maximum dry volume unit weight ($\gamma_{d,max}$) equal to 9.59 kN/m^3 were obtained.

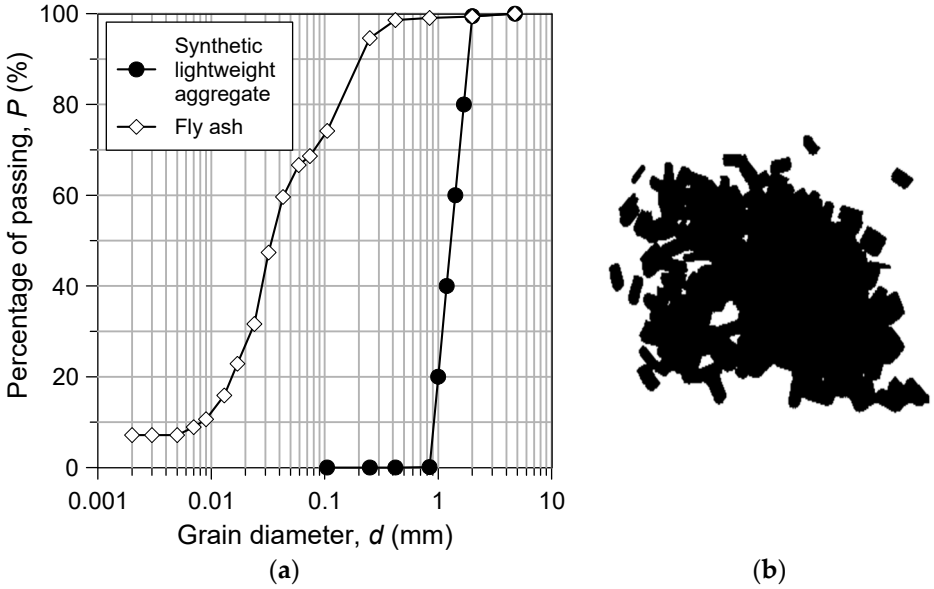
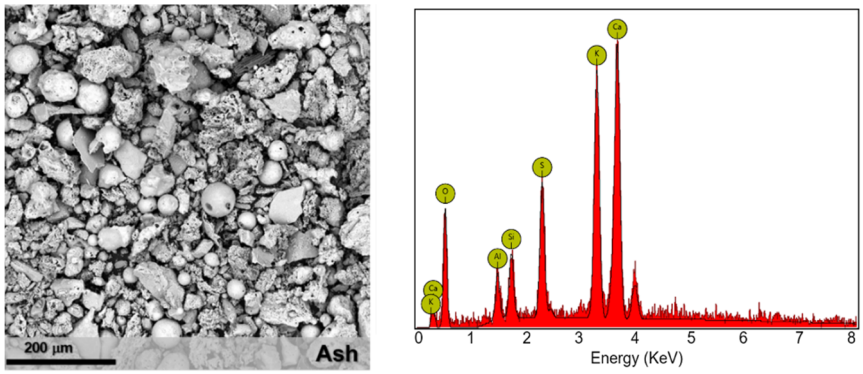


Figure 1. (**a**) Grading curves of tested materials and (**b**) picture of synthetic lightweight aggregates (SLAs).

A simple and fast system to gain information on the morphological aspect and on the crystalline structure of the fly ash resides in the use of microscopy and diffractometry, respectively. In particular, the SEM analysis showed a well graded grain size distribution and an irregular morphology (Figure 2a), mostly characterized as sub rounded to rounded particles. The chemical investigation was performed by the EDX analysis. Indeed, Ca, Si, Al, K, S and C (Figure 2b) were recognized as the main elements that, as evidenced by the complementary XRD analysis (Figure 3), were arranged in the form of well-defined calcium, aluminum and silicates crystalline phases. These structures were mainly identified as quartz (SiO_2), syngenite ($K_2Ca(SO_4)_2\ H_2O$), calcite ($CaCO_3$) and anorthite ($CaAl_2Si_2O_8$), in accordance with similar results reported for the fly ash derived from biomass combustion [32]. The total amount of calcium, arranged in different crystalline structures, was estimated by the EDX analysis equal to 10.9%. Therefore, the used biomass fly ash was identified as a class F fly ash, in accordance with other studies [5].



(**a**) (**b**)

Figure 2. (**a**) SEM micrograph of fly ash and (**b**) relative EDX analysis.

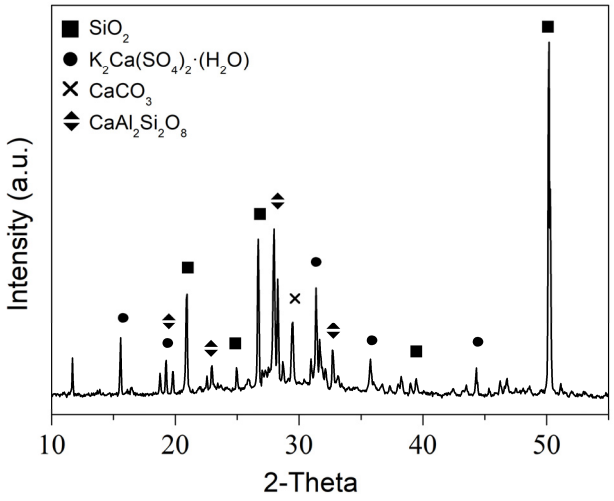


Figure 3. X-ray diffraction pattern recorded on fly ash sample.

The TGA-DSC analysis was used to study the behavior of ashes at different temperatures, highlighting their thermal stability up to 800 °C, without significant mass loss, attested around 8%.

The concentration of heavy metals (HMs) and potential toxic metals (PTMs), such as Cu, Ni, Zn, Mn and Fe, resulted clearly below the limit of concentration, in accordance with the reference limits [15].

The pH measure, being one of the most critical parameters for eco-compatibility and for landfill eligibility, recorded a value of 8.21, in accordance with the reference limits [15].

2.2. *Properties of Polymers*

For the synthesis of the aggregates, ashes from the biomass were mixed to a polymeric support, consisting of commercial high density polyethylene (HDPE). Polyethylene is the simplest, among synthetic polymers, and it is also the most common among plastic wastes. In this case, the use of TGA-DSC analysis on the pure polymer was necessary in order to evaluate its thermal stability during the extrusion manufacturing process. HDPE was stable up to 200 °C, as shown in Figure 4, and in accordance with the literature results [33].

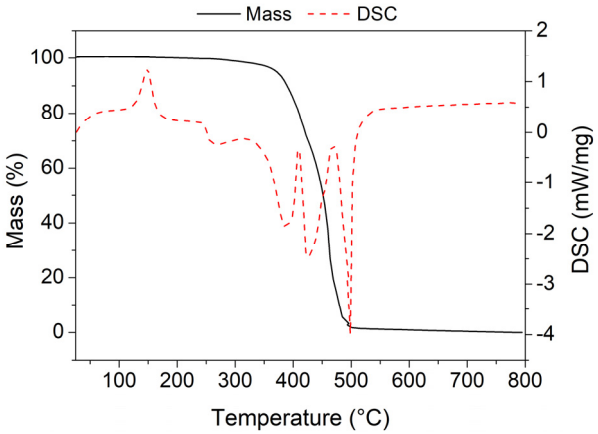


Figure 4. TGA-DSC analysis of pure polyethylene.

2.3. Manufacturing Process

The synthesis of new SLAs was carried out with a co-rotating twin-screw extruder, model FSCM15-40. Fly ash and HDPE pellets were homogeneously mixed together and extruded in a 1:1 weight ratio, considered to be the most suitable combination among the investigated ratios (1:4, 2:3, 3:2 and 4:1) to maximize the recycling of ash while simultaneously improving the extrusion process and preserving SLA's mechanical properties. A suitable procedure was developed as described below.

The mixture of fly ash and HDPE was fed through a hopper into a cylinder in which the polymer phase was melted at the established temperature of 200 °C, as suggested by TGA-DSC analysis, and dragged forward by the movement of a screw. In particular, inside the cylinder, the rotation of the extruder worm screw allowed the homogenization of the molten mass and its transport to the exit hole. Finally, the melt was forced to enter a die able to give the desired shape with no break in continuity. The matrix, generally shaped according to the desired profile, was, in this case a circular section for the production of 1.5 mm diameter wires. Then, the extruded product was solidified in a cooling area and finally cut into pellets of a previously established size, thanks to a cutter positioned in the final part of the equipment.

2.4. Physical and Chemical Properties of the Aggregates

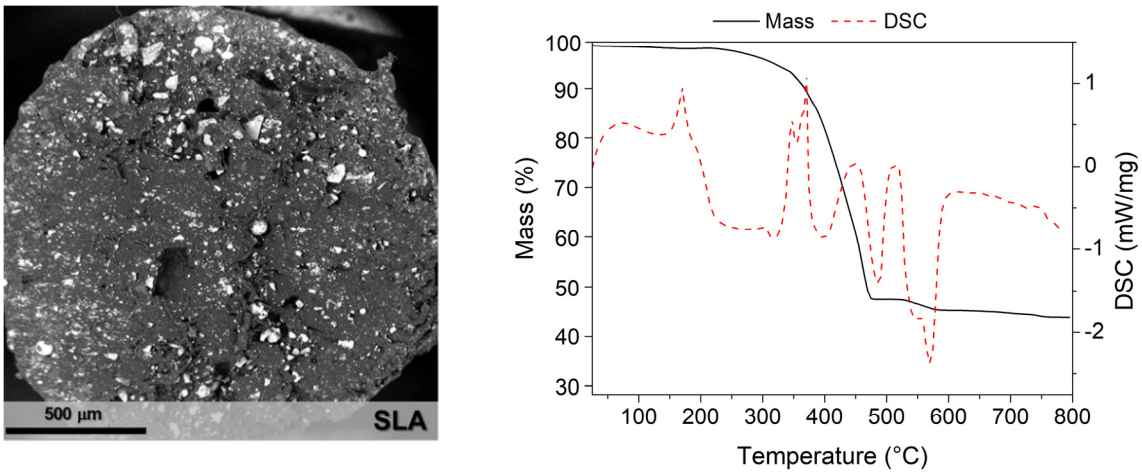
Figure 1a shows the particle size distribution curve of the new synthetic lightweight aggregates. As one can infer, the mean grain size (D_{50}) is 1.30 mm and the uniformity coefficient: $C_U = D_{60}/D_{10}$ is 1.54 where:

- D_{50} = sieve diameter corresponding to 50% of passing (by weight);
- D_{60} = sieve diameter corresponding to 60% of passing (by weight);
- D_{10} = sieve diameter corresponding to 10% of passing (by weight).

As regards the angularity of the aggregate granules, it was classified by analysing the particles with a visual comparison according to a sphericity and roundness chart [34]. This determined the synthetic lightweight aggregates particles to be sub-angular to angular (Figure 1b). Given the low value of C_U ($C_U < 2$), the material can be considered very uniform. For the determination of the specific gravity G_S of the aggregate granules, measurements with the hydrostatic balance [35] were carried out and a value of 9.8 kN/m^3 was determined. The maximum and the minimum dry density of the aggregate's masses were determined by using the downpipe pluvial deposition method [36] and the standard method provided by ASTM D4254 [37], respectively. The values obtained were $\gamma_{d,max}$ = 7.24 kN/m^3 and $\gamma_{d,min}$ = 6.05 kN/m^3. The small range of variation between these values is justified by physical features and particle size distribution of the aggregates.

In order to evaluate the morphological aspect of the new aggregates and to assess their thermal stability, SEM microscopy and TGA analysis were used, respectively. SLAs pellets showed a uniform

structure, both on the surface and in their cross section. Indeed, the ash results were uniformly distributed and well embedded in the HDPE matrix, as evidenced in Figure 5a. Thermal analysis showed the mass-loss behaviour of the new composite material as a function of temperature (Figure 5b). Common features in the shape of the two thermograms, SLAs and pure polymer, can be recognized. However, the presence of the ash determined a decrease in the thermal stability of the composite material when high temperatures were tested. In particular, in the aggregates, the onset temperature, referred to as the initial degradation, was lower than in the pure HDPE, 388.9 °C and 434.1 °C, respectively, in accordance with other results. This shows that the presence of an additive led to a thermally less stable matrix [33]. Moreover, as expected, a final residue, comparable with the nominal ash content, was also detected.



(**a**) (**b**)

Figure 5. (**a**) SEM micrograph of SLA cross section, (**b**) TGA-DSC curves of aggregates.

3. Test Results and Discussion

Laboratory tests were carried out on the new synthetic lightweight aggregates to verify their efficiency in removing heavy and potentially toxic metals, and, at the same time, in determining their mechanical and hydraulic characteristics. In particular, the experimental program included:

- batch leaching tests;
- incremental loading oedometer tests;
- direct shear tests;
- constant head permeability tests.

3.1. Batch Leaching Tests

Batch Leaching tests were carried out by using the UNI EN 14457-2 (Italian National Unification - European Committee for Standardization) [15] method (neutral conditions) as sketched in Figure 6.

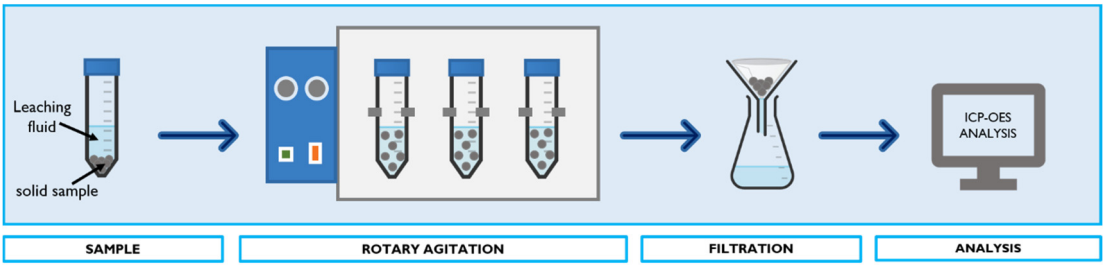


Figure 6. Schematic procedure of a batch leaching test.

By using the UNI EN 12457-2 method [15], 1.0 g of granulates was added with 10 mL deionized ultrapure water (sample to water 1:10) in a closed leak-proof bottle and shaken for different lengths of time (0.5, 1, 3, 6, 12, 24 h) at room temperature. Leachate samples were filtered through whatman WCN0.45mm membranes and analyzed with Inductively coupled plasma - optical emission spectrometry, ICP-OES, (Perkin–Elmer Optima 800, Waltham, MA, USA) for the metal quantifications in analogy with other reports [38].

Duplicates were performed for the test on three identical samples (blanks were prepared by the same sample treatment procedure) and the average value, having a deviation standard lower than 3%, was considered "con" Each ICP measurement was repeated 3 times and the average value for metal ion concentration (mg/L) was considered (Relative Standard Deviation, RSD, for all detected elements below 3%). Table 1 shows the concentration (ppm) of Cu, Ni, Zn, Mn and Fe released upon leaching tests of the fly ash and SLA samples. The limit value according to UNI EN 12457-2 [15] for the investigated metals is also presented for comparison purposes.

Table 1. Leaching of metals from fly ash and SLA samples under UNI EN 12457-2 [15] conditions.

Metal Ion	Limit Value	Fly Ash			SLA		
	(UNI EN 12457-2)	Concentration	Std. Dev.	RSD	Concentration	Std. Dev.	RSD
Cu^{2+}	0.05 mg/L	0.20 [mg/L]	0.028	1.67%	0.001	0.0001	2.89%
Ni^{2+}	0.01 mg/L	-	-	-	-	-	-
Zn^{2+}	3.00 mg/L	2.01 [mg/L]	0.094	0.50%	-	-	
Mn^{2+}	0.05 µg/L	0.50 [mg/L]	0.071	1.84%	0.002	0.0002	1.99%
Fe^{2+}	0.20 mg/L	2.90 [mg/L]	0.093	1.43%	0.2	0.015	1.19%

Iron, zinc and copper are released in significant amounts from fly ash (3 mg/L, 2 mg/L and 0.2 mg/L, respectively) under neutral conditions. Concentration of Cu and Fe registered are above the UNI EN 12457-2 [15] limit. At the same time, after 24 h, a negligible variation in pH is observed (pH = 8.1). On the other hand, the leaching tests of the SLA samples show a metal concentration that is significantly lower than that measured for fly ash and, most importantly, below the limit values.

As expected, the leaching of analyzed metals from fly ash and SLAs samples increased with time (Figure 7). Nickel was not detected for both fly ash and SLAs samples. In the latter, zinc was also not registered. However, it is worth noting that achieving desorption equilibrium occurs quickly for fly ash (60 min) as well as for SLA samples (30 min) (Figure 7).

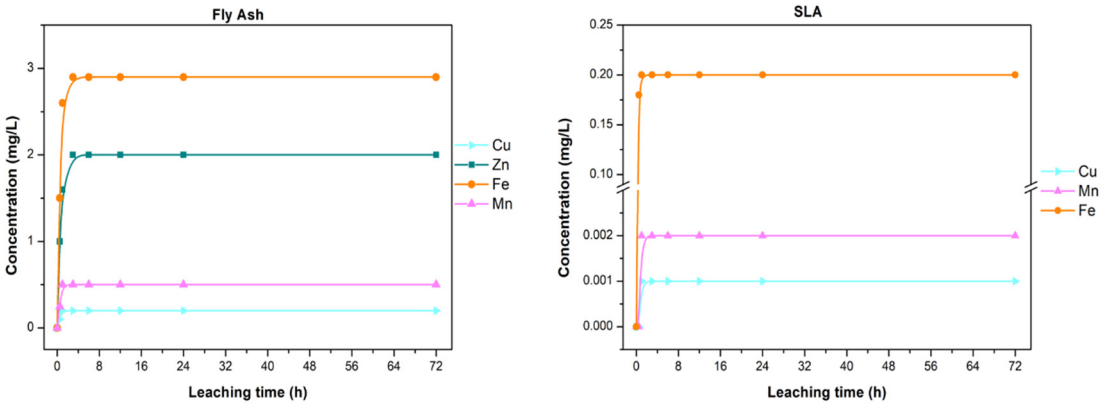


Figure 7. Metal concentrations trends during leaching tests from fly ash and SLA samples under UNI EN 12457-2 [15] conditions.

3.2. *One-Dimensional Compressibility Properties of the Aggregates from Oedometer Tests*

One-dimensional incremental loading compression tests ("oedometer tests") were performed on the SLAs in general accordance with ASTM D2435 [39] for assessing their compressibility features. Medium dense dry specimens, 20 mm in height and 71.4 mm in diameter, were reconstituted by the "tamping" method and subjected to incremental loads up to a maximum vertical stress of 2500 kPa. Each load increment was maintained for a time of 24 h during which the vertical displacements (ΔH) of the specimen were carefully tracked. Afterwards, specimens were subjected to a final unloading stage. Figure 8 shows, in semi-log scale, the plot of the vertical strains $\varepsilon_{v(24h)}$ versus the applied vertical normal stresses (σ'_{v0}), being $\varepsilon_v = \Delta H/H_0$, and H_0 the initial height of the sample.

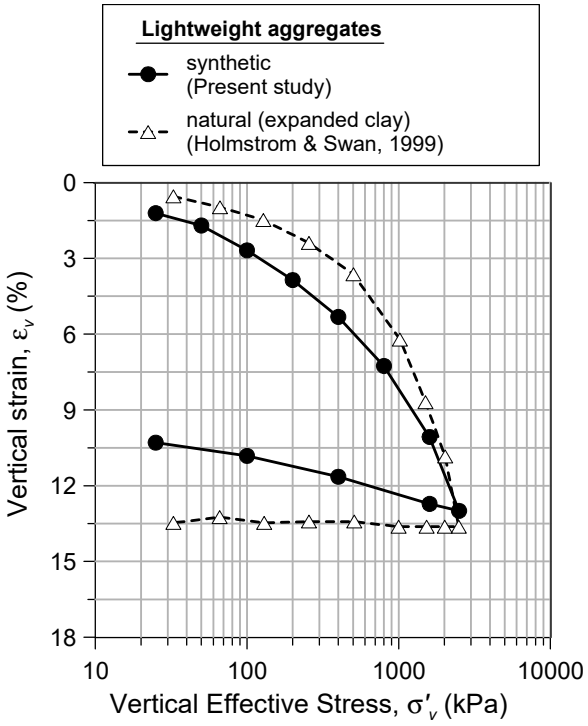


Figure 8. Vertical strain versus applied vertical stress for synthetic lightweight aggregates from one-dimensional compression tests.

The results, gathered from previous studies on lightweight natural aggregates [25], are also superimposed in the same figure. The aforementioned lightweight aggregates consisted of expanded clay tested at a density state similar to that investigated in the present research. Upon loading in the

range of the applied stresses, the comparison shows that there are no significant differences between the compressibility curves of the two aggregates. In particular the conventional expanded clay aggregates appear to be slightly stiffer than the newly developed SLAs in the very early portion of the curve while, at high applied stresses, these later exhibit a lower compressibility (evaluated in an incremental way) compared to the former ones. On the other hand, a relatively flat unloading response is displayed by the expanded clay, while a significant rebound is observed in the unloading stage of the synthetic aggregates manufactured in the present research. It is interesting to note that, after testing, the SLA specimen appeared compact and nearly "cemented" to the point that a forceful action was required to break the clusters of granules and to separate individual grains (Figure 9). To quantify whether any significant breakage of the individual SLA grains during compression occurred, the change of the grading characteristics of the sample before and after the oedometer test was evaluated. The gathered results did not evidence any appreciable change in the grain size distribution curve of the tested material before and after testing. Hence, the performance of the new lightweight synthetic aggregates in terms of crushing resistance, can be considered satisfactory, at least within the stress range adopted in the oedometer tests.

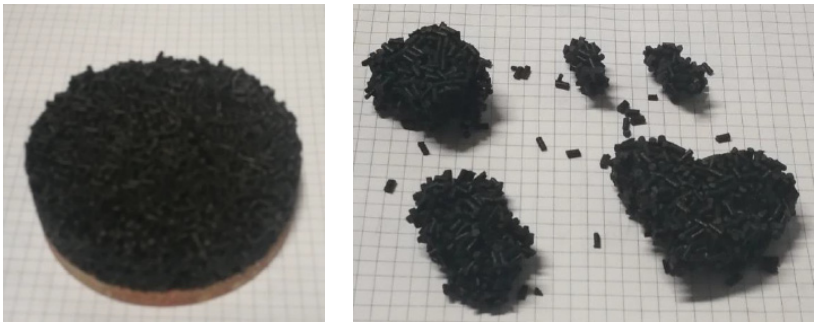


Figure 9. Pictures of the SLA specimen after one-dimensional compression tests (maximum applied vertical stress = 2500 kPa).

In order to investigate the "creep behavior" of the new lightweight synthetic aggregates under one-dimensional compression, two loading increments of the oedometer test were maintained for a longer time (48 h). The vertical stresses of the aforementioned "creep steps" were respectively: 400 kPa and 800 kPa. A creep coefficient (C), which denotes, in a logarithmic time scale, the rate of change of the vertical strains with time (t), was determined for both of them. C is defined by the following expression:

$$C = \Delta\varepsilon_v/\Delta\log t \quad (1)$$

and is evaluated from the final linear portion of the ε_v-log(t) curve. Calculations carried out for the two aforementioned stress levels (400 kPa and 800 kPa) provided the following values: $C = 4.2 \cdot 10^{-3}$ and $C = 6.4 \cdot 10^{-3}$, respectively. The results obtained suffer the shortcoming of a relatively short observational time (48 h). For this reason, they need to receive further confirmation by future investigations based on longer observational periods.

3.3. Shear Strength Properties of the Aggregates from Direct Shear Tests

In order to assess shear strength properties of SLAs manufactured in the present research, direct shear tests were performed on dry cylindrical specimens 63.5 mm in diameter and 20 mm in height. SLA specimens were reconstituted by using the tamping method at a medium dense state and consolidated at different values of effective vertical stress: σ'_{v0} = 50, 70, 100, 200 kPa. In the consolidation stage each loading step was maintained for about 24 h; throughout this time changes of sample height were measured to verify that any creep displacement at the end of each loading step was practically exhausted.

The shearing phase was carried out at a constant rate of horizontal displacement equal to 0.06 mm/min in all the tests. In this phase both the vertical and horizontal forces as well as the displacements in two directions were automatically registered and recorded.

The results of the direct shear tests for each vertical consolidation stress are shown in Figure 10. In particular: (i) plots of the shear stress (τ) against the horizontal displacement, *s* (mm) (Figure 10a); (ii) plots of the vertical displacement *v* (mm) against the horizontal displacement *s* (mm) (Figure 10b) are reported. As it can be seen from Figure 10b, SLAs evidence a predominantly dilating behavior with a constant (or slightly increasing) rate of dilation (i.e., slope of the curves) in the final stage of the test. Correspondingly, the shear stress–displacement curves in Figure 10a are characterized by a continuous increase in the mobilized shear stresses without reaching a peak. Furthermore, the observed volumetric response (dilation rate) under different effective vertical stresses is consistent with what is expected for granular soils in the sense that the dilation rate tends to increase with the decrease in the effective vertical stress.

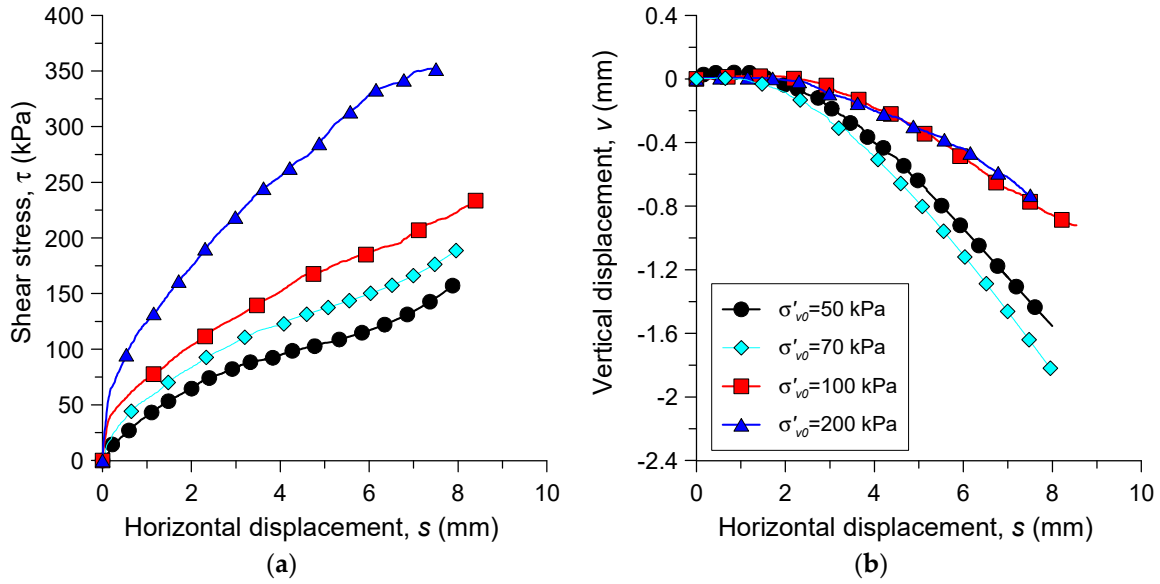


Figure 10. Direct shear test results on synthetic lightweight aggregates in terms of (**a**) shear stress versus horizontal displacement and (**b**) vertical displacement versus horizontal displacement (σ'_{v0} = 50, 70, 100, 200 kPa).

Due to the peculiar features of the stress-strain behavior mentioned above, a limiting displacement approach was adopted to define failure conditions of the investigated SLAs. A conventional horizontal displacement (s) of 7.5 mm, i.e., about 12% of the sample diameter (D), was assumed to mark the "peak failure" in all tests. "Peak failure" data of the SLAs, plotted in the τ_{max}-σ'_{v0} plane, are shown in Figure 11a. The linear regression through the experimental data leads to an internal peak friction angle φ′ = 54° and a cohesion intercept c′ = 79.5 kPa. However, due to the granular nature of the investigated SLAs, the peak strength envelope is more likely to be curved with a pronounced curvature at very small vertical stresses σ'_{v0}; interpreting the failure data in terms of purely frictional behavior that would imply a dependence of the secant "peak" friction angle, φ_s' on the effective vertical stresses. Figure 11b evidences the trend of φ_s' with σ'_{v0} evaluated for the investigated SLAs. Similar results have been reported by Holmstrom and Swan [25] from direct shear tests carried out on medium dense specimens of light expanded clay aggregates.

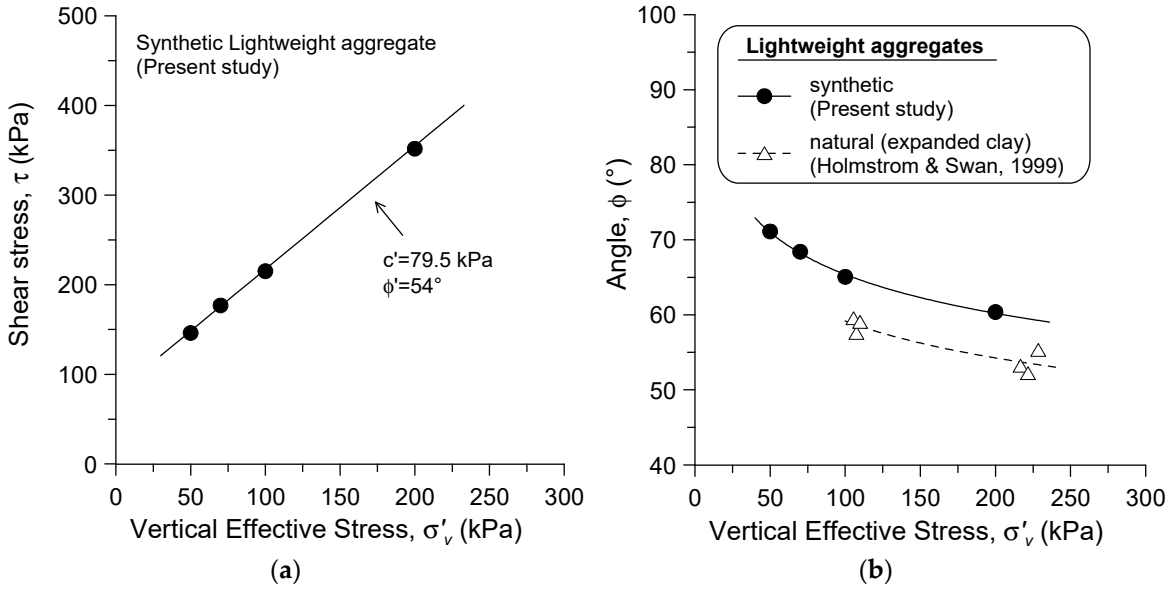


Figure 11. (**a**) Peak failure envelopes obtained by means of direct shear tests performed on synthetic lightweight aggregates and (**b**) comparison between internal peak friction angle obtained in the present study and literature data on natural lightweight aggregates.

In Figure 11b data reported on [25], are superimposed on those gathered in the present research; the comparison evidences a very good agreement, the values of SLAs being just a little higher than those provided by the lightweight expanded clay ones. As a final remark it is worth mentioning that one of the main concerns of the direct shear tests conducted on granular materials (such as the aggregates) is the size of the experimental box adopted for the tests compared with the dimensions of the grains. Using a small size box can induce scale effects on test results. As a practical rule [40], when the L/D_{50} ratio between the size (L) of the box and the mean grain size (D_{50}) of the aggregates is ≥ 50, test results are not affected by scale effects. In the present study this ratio was slightly lower than 50 ($\cong 45$) and for this reason size effects can be considered negligible.

3.4. Water Absorption and Hydraulic Properties of the Aggregates

Water absorption is the main concern when it leads to artificial aggregates, as it can be high for most of the aggregates. Furthermore, water absorption gives an idea on the internal structure of the aggregates: aggregates having more absorption are generally more porous.

The procedure for the measurement of water absorption was conducted on SLAs with reference to the UNI EN 1097-6 method [41]. The test provides water absorption on the sample as function of time. A SLA sample of known weight (W_2 = Weight of oven dry aggregates in air) was submerged in water for a prefixed time and afterwards the weight of the saturated aggregates in air (W_1) was also measured. The ratio $[(W_1 - W_2)\cdot 100]/W_2]$ is the water absorption coefficient W (%).

The results in terms of the water absorption coefficient against time are plotted in Figure 12a: a rapid trend towards imbibition, with values of absorption coefficient over time around 20%, can be observed. The coefficient W was equal to 16.3% after 24 h and resulted 19.3% after 30 days.

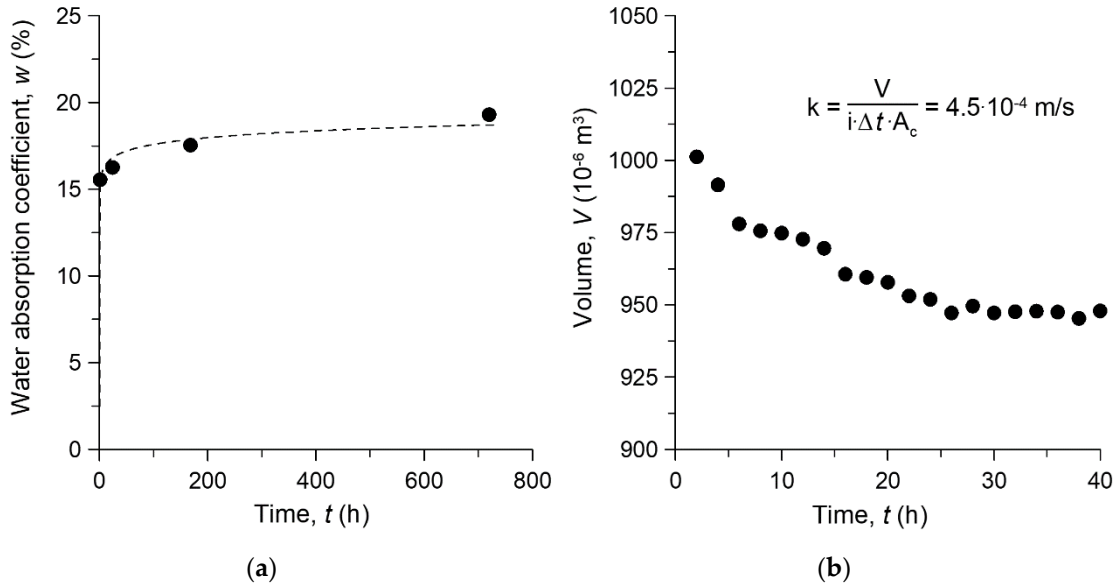


Figure 12. (**a**) Water absorption features and (**b**) measurements of hydraulic conductivity under constant head (Δt = 2 min) of SLAs tested in the present study.

In order to investigate permeability features of SLAs, tested in the present study, measurements of hydraulic conductivity under constant head, were performed following the procedure suggested by the ASTM D2434 [42]. Specimens of synthetic lightweight aggregates (93 mm in diameter) having the desired density (D_R = 66%), were prepared by dry tamping in five layers (each one being h = 65 mm in height). Preliminary checks were conducted to verify that the adopted preparation procedure leads to samples of satisfactory uniformity. Afterwards, specimens were subjected to a pre-saturation phase by allowing de-aerated water to flow in the sample ("flushing") for an adequate length of time.

During the test, the specimen was maintained at "constant hydraulic head" and the volumes of water (V) coming from the cell at constant intervals of time (Δt), were measured as long as a stabilization condition was reached. Such measurements were plotted against the cumulative time (t), as shown in Figure 12b, where a time step of 2 min was adopted. The curve, reported in this figure, displays an approximately regular trend towards a final stabilization around a value of $V = 945 \cdot 10^{-6}$ m^3 and this final value of V was used in the calculations for determining the hydraulic conductivity (K). K was computed through the following expression derived from Darcy's law:

$$K = \frac{V}{i \cdot \Delta t \cdot A_c} \quad (2)$$

where:

- A_c = cross-sectional area of the sample;
- i = hydraulic gradient;
- V and Δt have the meaning specified above.

The calculations provided a value of K= $4.5 \cdot 10^{-4}$ m/s.

4. Conclusions

The paper presents preliminary results on the synthesis, characterization and tests, carried out to verify the suitability of innovative synthetic lightweight aggregates (SLA) for geotechnical engineering applications (i.e., lightweight embankments, re-profiling of slopes, backfill against foundations, retaining walls, drain trenches in slopes, pavements systems, as well as vibration isolation, etc.). The aggregates were manufactured by a thermal process, combining and co-extruding plastic and biomass

fly ash. The results, displayed in the present work, refer to aggregates with a 1:1 fly ash/HDPE ratio by weight.

The first phase was carried out for the development of an adequate methodology and manufacturing technique for the new aggregates. Subsequently, SLAs were subjected to laboratory testing and specific analysis for assessing: (a) physical–chemical properties (i.e., chemical composition, scanning electron microscopy analysis, mineral leachability, water absorption, specific gravity, grain size distribution); (b) permeability features; (c) mechanical properties (one-dimensional compression and shear strength behavior).

The main conclusions which can be drawn from the results are as follows:

- The morphological and structural analysis of the new SLAs pointed out the crystallographic nature of the utilized fly ash and displayed their homogeneous distribution in the polymeric matrix. Ash grains were well embedded in HDPE and stable in their aggregate configuration. The specific gravity of the SLA granules was approximately 9.8 kN/m^3.
- The leaching tests of SLA samples showed that the metal ions concentration (Cu^{2+}, Ni^{2+}, Zn^{2+}, Mn^{2+} and Fe^{2+}) was significantly lower than that measured for fly ash and, most importantly, was below the limit values (UNI EN 12457-2 method [15]).
- Oedometer tests performed on the new lightweight synthetic aggregates for assessing their compressibility features evidenced that the behavior was consistent with that observed in the literature on lightweight aggregates of different nature (natural and synthetic). Additionally, no appreciable grain crushing phenomena or degradation of SLAs, at higher vertical stresses, were found at the end of the tests.
- Direct shear tests performed on the SLAs evidenced, for any effective vertical stress, a stress–strain behavior characterized by a continuous increase in the mobilized shear stresses without reaching a distinct peak; for this reason a strength criterion based on the assumption of a limiting displacement ratio, s/D = 12%, was assumed.
- The "peak strength" envelope obtained following this approach exhibited a cohesive-frictional strength behavior; its linearization, in the considered stress range (σ'_{v0} = 50 to 200 kPa), provided the following strength parameters: $\Phi' = 54°$ and $c' = 79.5$ kPa. These values compare well with those obtained by other researchers on both natural and synthetic lightweight aggregates.
- The water absorption tests performed on the new lightweight synthetic aggregates showed a rapid trend to the imbibition of the SLAs with measured values of the absorption coefficient which stabilized over time, at around 20%.
- The hydraulic characteristics of the aggregates were investigated by means of constant head permeability tests; these tests provided a value of the hydraulic conductivity (K) equal to $4.5 \cdot 10^{-4}$ m/s.
- When the results, gathered from the SLAs manufactured in this research, are compared with those obtained in similar research, concerning natural and synthetic aggregates, they appear promising for the potential utilization in geotechnical engineering. However, further insights into the effects of different proportions of the components in the mixture (recycled plastic and biomass fly ash) are needed as well as their behavior under cycling loading.

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