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Characterization of lightweight aggregates produced with clayey diatomite rocks originating from Greece

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Abstract

A clayey diatomite rock originating from Greece, mainly composed of opal-A, smectite and vermiculite was tested for the production of lightweight aggregates (LWAs) by mixing the raw material with 2–5% sawdust. The mixtures were pelletized, dried at 100 °C and burnt to 1100 °C. The laboratory LWA had strength and density similar to commercial LWAs originating from Germany and Denmark. The porosity of their mantle was lower than that of the inner part. Pores were abundant, ranging from irregular to spherical and from 2 to 500 μm in size, resulting in low apparent density values. The texture of the Greek and German LWA was clearly amorphous, whereas subhedral crystal assemblages were detected in the interior of the Danish LWAs. LWA produced by the Greek clayey diatomite rocks can be utilized for the production of LWA on an industrial scale.

Keywords: Lightweight aggregates; Scanning electron microscopy; Clayey diatomite rocks; Greece

1. Introduction

Clayey diatomite rocks have been located in the uppermost levels of thick Tertiary, lacustrine deposits located in central Greece containing, besides opal-A (diatom frustules), substantial amounts of smectite and vermiculite [1,2]. Because of their nature and their similarity with sedimentary rocks of Denmark used for the production of lightweight aggregates (LWAs), absorbents and insulation units, the Greek clayey material was tested for its suitability as LWA raw material.

LWAs of natural and artificial origin are currently used in lightweight structural concrete, in precast structural units, road surfacing materials, plaster aggregates, loose insulating fill, gardening and hydroponics, in geotechnical applications and in thermal and sound insulation [3–10].

In most European countries, the production of artificial LWA is mainly based on the burning of clayey rocks that contain expanded clays, such as smectite and vermiculite, commonly using crude oil or sawdust as burnable material [4,7,11]. The clayey material is usually carbonate minerals free, although a small

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quantity of dolomite is sometimes added in the mixture, because of its property to act as a flux agent.

The aim of the present paper is to characterize and test clay and amorphous silica-rich rocks and their sintered products for the production of LWA on a laboratory scale.

2. Fieldwork and bulk rock sampling

During some reconnaissance geological fieldwork in Tertiary sedimentary basins of central Greece, several clayey diatomite rock samples were extracted from a thick clayey sedimentary succession. The preliminary laboratory analysis revealed that the clayey material is chemically and mineralogically homogeneous throughout the Upper Miocene succession [1]. It was, therefore, decided to collect two bulk rock samples from two distal parts of the basin, weighing approximately 100 kg each for their full characterization and testing procedures. The bulk rock samples were low in density and friable, having a brownish yellow colour and exhibiting thin lamination.

3. Experimental procedures

The analyses and the experimental procedures included the following: crushing, homogenisation and characterization of raw materials, including mineralogical semiquantitative XRD analysis based on the middle of the height peak, interpretation based on the software EWA2.2 (XRD, SIEMENS D5000, 40 kV, 40 mA, 1°/min, Cu–Ka radiation), chemical analysis (XRF) of the oxides: Na_2O, K_2O, CaO, MgO, Fe_2O_3t, Al_2O_3 and SiO_2 (XRF, PHILIPS PW1010, 50 kV, 50 mA) and scanning electron microscopy (SEM) analysis (JEOL-JSM 5600 20 kV, 0.5 nA, time of analysis 50 s) and PHILIPS XL30 E-SEM 30 kV, 20 mA).

Table 1
XRD mineralogical analysis of the Greek clayey diatomite raw material

Samples	Qtz	Ver	Sm	OA[a]	Ill	Fl	Chl
GRE-1	MD	MJ	MJ	MJ	TR	MD	MD
GRE-3	MD	MJ	MJ	MJ	TR	MD	MD

Qtz = quartz, Ver = vermiculite, Sm = smectite, OA = opal-A, Ill = illite, Fl = feldspar and Chl = chlorite. MJ = major component, MD = medium component and TR = minor trace component.
[a] Opal-A was mainly identified by SEM analysis.

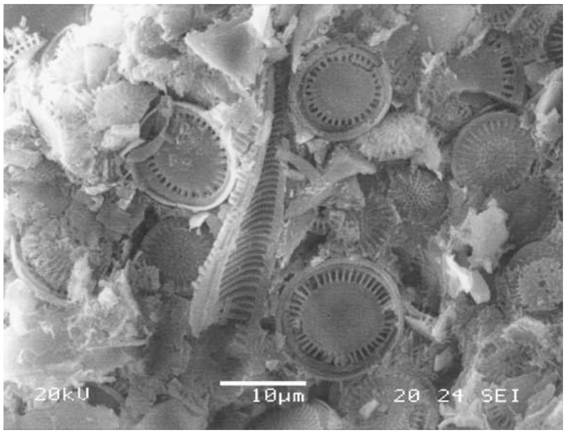

Fig. 1. Disc-shaped and boat-like diatom frustules hosted in a clayey matrix from the Greek raw material.

Laboratory production of LWAs used the Greek clayey materials and sawdust as combustible additive. The procedure included pulverising of the clayey diatomite raw material weighing approximately 20 kg of less than 100 μm in size, mixing with 2%, 3.5% and 5% sawdust with particles of less than 1 mm in size, homogenising and mixing with water forming pellets 5–20 mm in size. The added water/sawdust–clayey rock mixture ratio was < 1/2 to approximately 1/2, to have the same workability during the manufacturing of the LWAs. A series of trials was performed with constant addition of water to the solid clayey diatomite–sawdust mixture ratio (w/s) at 1/2 (see Fig. 8).

The formulated pellets, sizing of 5–20 mm, were dried at 100°C in an oven for 24 h.

The pellets produced were burnt in a laboratory kiln for 12–15 min at 1100 °C (see Fig. 3).

The texture and porosity of the mantle and the core of several pellets, as well as their chemical composition was measured by using SEM and microprobe analysis (EDX, OXFORD LINK ISIS 300, software package ZAF correction) of the laboratory-produced Greek LWA and commercial LWA derived from Germany and Denmark.

Testing and comparison was made of the sintered products produced in the laboratory with the German and Danish LWA. The fracture load of a series of single pellets was measured by pressing them until they cracked in the press apparatus Tonitechnic/ Toninorm.

The results are presented in Tables 1–5 and Figs. 1–8.

4. Results and discussion

4.1. Raw materials characterization

4.1.1. Mineralogical analysis

The XRD analyses of the two bulk rock samples are presented in Table 1. The samples are characterized as clayey diatomite, as these are mainly composed of amorphous silica (opal-A) that has biogenic origin mainly represented by diatom frustules and rarely by sponge spiqules, and smectite and vermiculite. Other clay minerals, such as chlorite and illite, are also present in variable amounts. Detrital minerals, such as feldspar and quartz, are also important constituents, whereas carbonate minerals are absent.

4.1.2. Chemical analysis

The chemical analyses of the bulk samples are shown in Table 2. Silica, alumina and iron oxide were the main constituents of the samples. The SiO_2 content corresponds to both diatomaceous silica and aluminosilicate minerals present in the samples, Al_2O_3 to aluminosilicate minerals, and Fe_2O_3t to the high amounts of chlorite and vermiculite present in the samples. The CaO and MgO contents are low due to the absence of carbonate minerals. Both are associated with the presence of Ca-smectite, whereas CaO may correspond to the presence of subsidiary Na–Ca feldspars. The loss on ignition (LOI) of the samples is mainly attributed to loss of the H_2O contained in clay minerals and the SiO_2*nH_2O that is the mineral phase of the diatom frustules. The Na_2O and K_2O content are mainly attributed to the presence of feldspars and illite.

Table 2
XRF chemical analysis of the Greek clayey diatomite bulk rock samples

	GRE-1 (%)	GRE-3 (%)
Na_2O	1.28	0.48
K_2O	2.58	1.92
CaO	1.82	1.35
MgO	1.79	1.81
MnO	n.a.	n.a.
Fe_2O_3t	8.08	5.90
TiO_2	n.a.	n.a.
Al_2O_3	17.83	16.68
SiO_2	59.52	64.04
P_2O_5	n.a.	n.a.
LOI	7.37	8.06
Total	100.27	100.20

n.a. = not analysed.

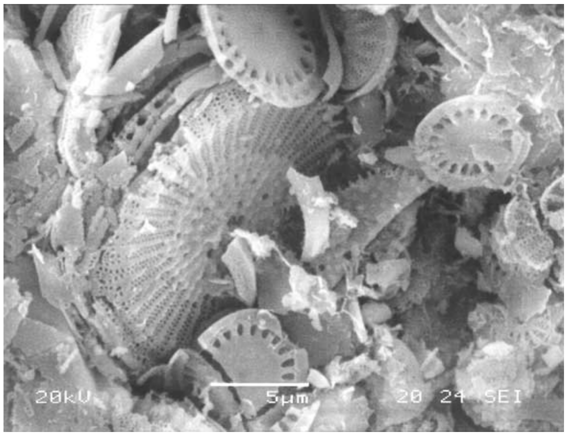

Fig. 2. Disc-shaped diatom frustules hosted in a clayey matrix from the Greek raw material.

4.1.3. SEM analysis

The SEM study of the diatomaceous rocks showed that the diatom frustules were mostly well preserved, having cylindrical or disk shape and ranged in size from 5 to 30 μm (Figs. 1 and 2). The predominant shape of the diatoms in the entire clayey diatomite succession is that of disk, belonging to the *Cyclotella* sp. (Fig. 1). The degree of preservation of the diatoms' frustules minute structure was good. The frustules commonly form assemblages of random orientation, hosted in the clayey groundmass.

4.2. Characterization of sintered products

4.2.1. LWA macroscopic characteristics

Concerning the laboratory-produced LWAs, they mostly retained their original shape that was predominantly spherical, having a red brown homogeneous colour throughout the LWA surface. Cauliflower-like pellets were rarely formed. Laboratory investigations on the behaviour of the laboratory LWAs during their thermal treatment revealed a significant degree of expansion of some pellets, especially the largest ones. Visible by naked eye pores or fissures on the mantle of these pellets rarely occur. (Fig. 3)

By contrast, the Danish LWAs are mostly asymmetric with irregular plate y, spherical or ellipsoidal shape with pores of less than 2 mm in size that commonly reached the surface of the pellets. Their colour is grey to grey black. The German LWAs are almost spherical in shape having a reddish colour. The pore development on the surface of the LWAs is intermediate between the Greek and the Danish LWAs.

4.2.2. Mineralogy and chemistry of the LWAs

The laboratory-produced LWA and the commercial LWAs were examined by XRD to identify the synthetic mineral phases formed during sintering. The results are presented in Table 3.

The mineralogy of the Greek and the German and Danish LWAs is characterized by the presence of hercynite ($FeAl_2O_4$) and mullite ($Al_6Si_2O_{13}$; Table 3). Hematite (Fe_2O_3) is also present in all samples, whereas magnetite (Fe_3O_4) was detected in the German sample. Another synthetic mineral phase detected in only the Danish and German samples is magnesium aluminium iron oxide ($MgFeAlO_4$). The absence of this mineral from the Greek LWAs is attributed to the absence of Mg- or Ca/Mg-rich minerals from the raw materials mineralogy. Sodium and potassium feldspars, such as albite and sanidine, and quartz are present in all samples, probably reflecting relics of the primary mineralogy of the raw materials.

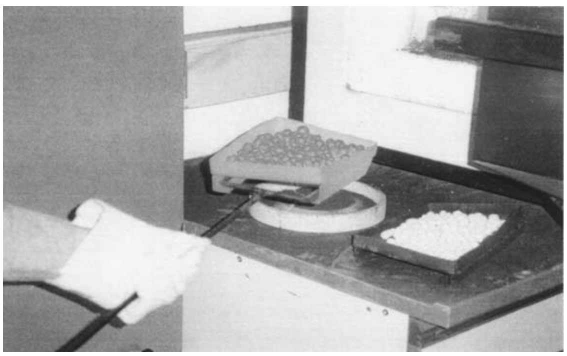

Fig. 3. Laboratory production of LWA before (right) and after burning (left).

Table 3
XRD analyses of the laboratory (GRE) and the commercial LWAs (DEN and GER)

LWA	Gl	Qtz	Feld	Herc	Mull	Mgnt	Hem	$MgFeAlO_4$
GER	MJ	MJ	TR	MD	MD	TR	TR	TR
DEN	MJ	MJ	MD	MD	MD	MD	TR	TR
GRE	MJ	MJ	MD	MD	MD	–	TR	–

GER = German LWA, DEN = Danish LWA and GRE = Greek LWA. Gl = amorphous phase, Qtz = quartz, Feld = feldspars (albite and sanidine), Herc = hercynite, Mull = mullite, Mgnt = magnetite and Hem = hematite. MJ, MD and TR are the same as (defined) in Table 1.

Despite the presence of the crystalline mineral phases above, the major constituent of all samples is the amorphous, glassy phase that is more prominent in the Greek LWAs (Table 3).

Microprobe analysis of the three categories of the LWAs compared in this study revealed some dissimilarities in their major element content probably due to the different raw materials used by the various producers and hence, differences in the nature of the glassy phase which predominates in all three samples (Table 4). In general, the Danish samples are richer in iron, the German samples are richer in alumina and the Greek samples are richer in silica; the latter probably due to the high amounts of the diatomaceous silica of the raw materials.

4.2.3. SEM determinations on the LWA structure

To measure and compare the porosity, the pore size and the internal and external shape of the laboratory

Table 4
Average values of microprobe analysis of the matrix of the LWAs studied

	GRE	GER	DEN
Na_2O	1.52	1.28	0.24
K_2O	2.20	3.60	2.40
CaO	3.24	1.46	6.54
MgO	1.77	1.54	4.52
MnO	BDL	BDL	BDL
Fe_2O_3t	12.34	22.16	32.90
TiO_2	BDL	BDL	2.19
Al_2O_3	18.23	26.18	15.38
SiO_2	60.82	44.10	35.76
P_2O_5	BDL	BDL	BDL
Total	100.12	100.32	99.93

Samples GRE, GER and DEN are the same as (defined) in Table 3. BDL = below detection limit (0.10%).

and the commercial LWAs, broken chips of various pellets were used. It was found that the porosity of the LWA's core varied, resulting in apparent density dissimilarities (Figs. 4, 5 and 6). The pore size of the LWAs ranged from 2 to 500 μm, with the larger pores being observed in the Danish ones. The shape of the pores varied from irregular to almost round. They exhibit similar internal and external texture with LECA commercial products [12], as well as LWAs used by Faust [13] in producing LWAC.

The external surface of the Greek LWAs is practically with no pores, whereas inwards, there is a progressive development of larger pores that have a honeycomb shape (Fig. 4, uppermost two and lowermost left). Rarely, almost spherical "drops" are developed in the interior of the pellets, having the same chemistry as the growing media (Fig. 4, lowermost right). Although the Greek raw material contained significant amounts of disk-shaped and cylindrical diatom frustules, no relics of these were detected in the sintered products. This could be explained by the dissolution of the amorphous silica at temperatures of about 1100 °C and the participation of the dissolved silica in the formation of the synthetic Fe–Si, Al–Si minerals detected in the LWAs.

The German LWAs have more porous external surfaces than the Greek ones (Fig. 5, uppermost two). Most of the pores have spherical shape of various sizes and are interconnected via microchannels (Fig. 5, uppermost right, lowermost left).

The Danish LWAs have much larger and irregular pores by comparison with the other two groups

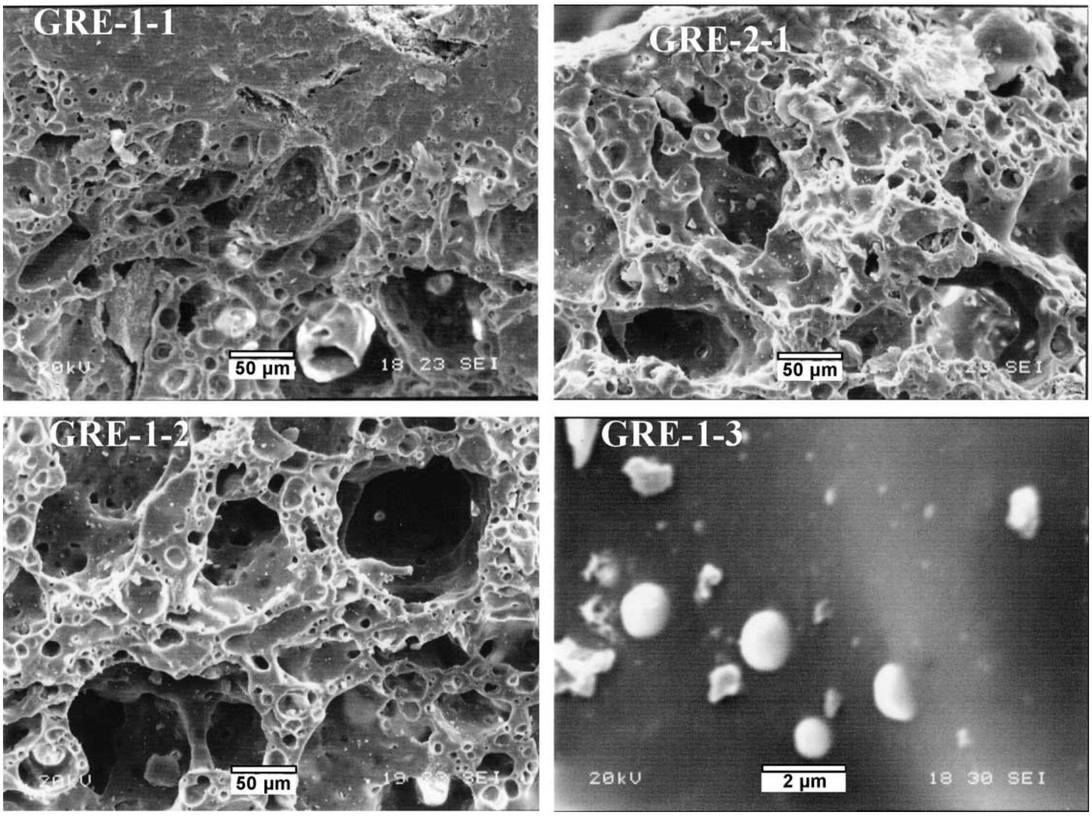

Fig. 4. SEM images of the laboratory-produced Greek LWAs. Uppermost left: the thin, almost massive mantle of the laboratory Greek LWAs is turning to porous matrix inwards. Uppermost right: pore systems close to the surface of the LWAs that form honeycomb channels. Lowermost left: the pores in the interior of the LWAs have various sizes and shapes and are interconnected, via small channels. Lowermost right: ultrafine Si–Al–Fe–rich "drops" locally grown in the interior of a pore.

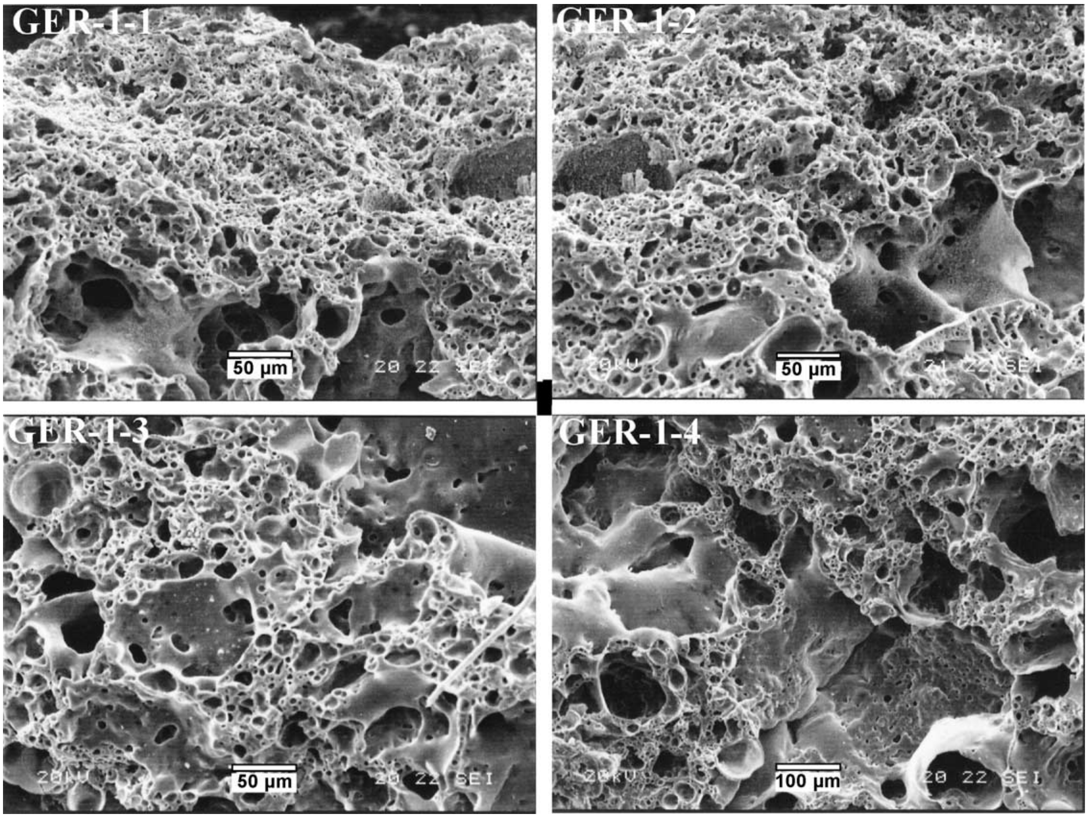

Fig. 5. SEM images of the commercial German LWAs. Uppermost left: the microporous mantle of the German LWAs is turning to more porous matrix inwards. Uppermost right: pore systems close to the surface of the LWAs that are interconnected with microchannels. Lowermost left: the pores in the interior of the LWAs have various sizes and shapes and are interconnected, via small channels. Lowermost right: the smaller pores in the interior of the LWAs are spherical, whereas the bigger ones became asymmetric.

(Fig. 6). The pore system of the external surface of this group is higher than the other two LWAs. By contrast to the other two LWA groups, the presence of open pores ejected to the surface of the Danish LWAs is common.

Whereas almost all samples studied have a "glassy" appearance, subhedral crystal assemblages were detected in the interior of some of the Danish LWA (Fig. 6, lowermost right).

4.3. Testing and comparison of the laboratory-produced and commercial LWA

4.3.1. Apparent density

The apparent density of the laboratory and the commercial LWAs was measured after grouping the pellets of three LWAs samples into three size groups, namely, 5–10, 10–15 and 15–20 mm. The results are given in Table 5.

After grouping of the commercial and the laboratory-produced LWAs in three ranges of diameter size 5–10, 10–15 (both with constant or variable water/solid ratio) and 15–20 mm, the apparent density of 2000 ml of each group was measured. In general, the LWAs with the largest diameter appeared with the lowest apparent density that is ranging between 0.55 and 0.79 g/cm^3 (Table 5). This can be attributed to the better ability of the larger pellets to develop large pores, and hence lower apparent density values during sintering.

Comparing the same group of the laboratory LWAs which have a diameter 10–15 mm but formed with variable (< 1/2) and constant (1/2) water/solid ratio, it was shown that the apparent density of the second

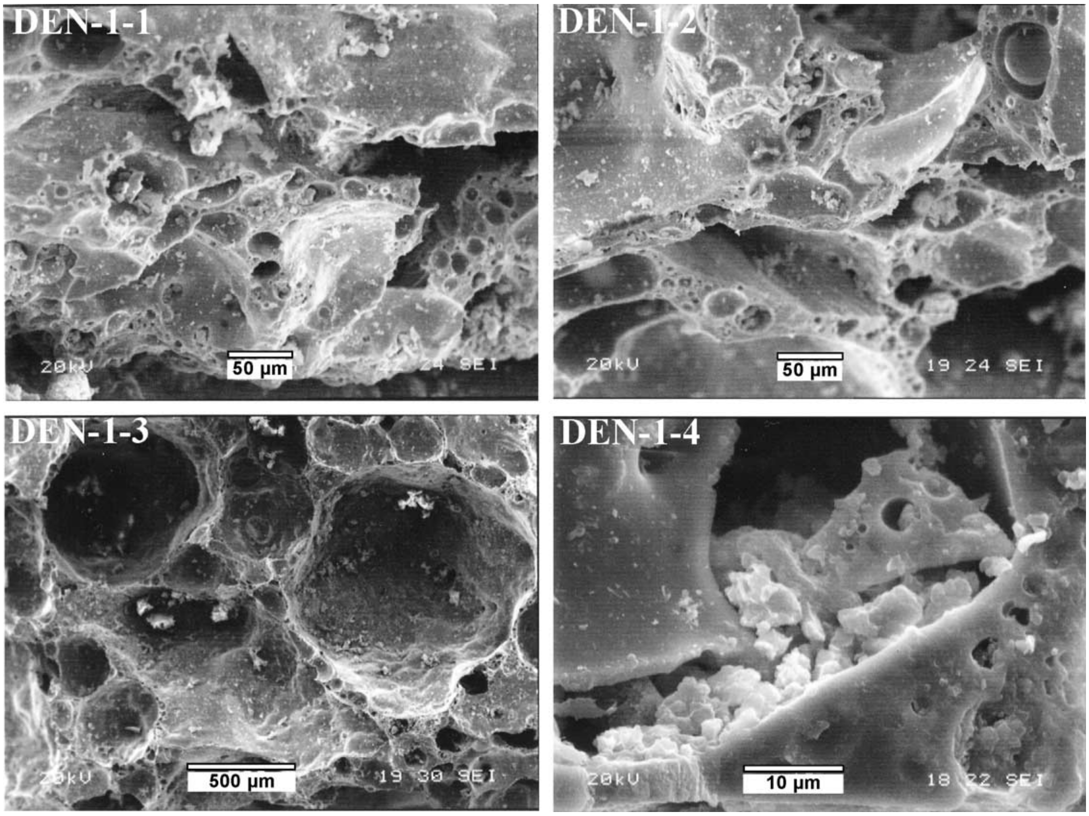

Fig. 6. SEM images of the commercial Danish LWAs. Uppermost left: the mantle of the Danish LWAs is the most porous among the three groups of LWAs studied. Open pores are visible throughout the mantle of the LWAs. Uppermost right: pore systems composed of spherical small and large "platy" pores close to the surface of the LWAs. Lowermost left: the pores in the interior of the LWAs are the largest among the samples studied, having a subspherical shape. Lowermost right: subhedral fine-grained Si–Al–Fe-rich crystals locally grown in the interior of pores in the Danish LWAs.

group was lower (Table 5). The differences are larger in the LWAs formed with 5% sawdust. This can be attributed to the better wetting and expansion of sawdust, before drying the LWAs.

4.3.2. Fracture load

One litre of pellets of each LWA's size and origin underwent a mechanical shatter by using the press instrument described in the experimental techniques

Table 5
Diameter, apparent density and fracture load of laboratory-produced and commercial LWAs

	$d = 5–10$ mm		$d = 10–15$ mm		$d = 10–15$ mm (w/s = 1/2)		$d = 15–20$ mm	
	N (g)	ρ (g/cm^3)	N (g)	ρ (g/cm^3)	N (g)	ρ (g/cm^3)	N (g)	ρ (g/cm^3)
GRE-A	416	0.58	690	0.68	451	0.53	560	0.54
GRE-B	454	0.59	536	0.56	503	0.54	551	0.56
GRE-C	659	0.74	2029	0.93	736	0.69	1190	0.79
GER	747	0.68	986	0.73			1205	0.79
DEN	466	0.75	580	0.65			524	0.55

N = fracture load, d = diameter and ρ = apparent density of the LWA. GRE-A: LWA with 2% sawdust, GRE-B: with 3.5% sawdust and GRE-C: with 5% sawdust. GER: German LWA and DEN: Danish LWA. w/s corresponds to the added water/solid ratio that was constantly 1/2.

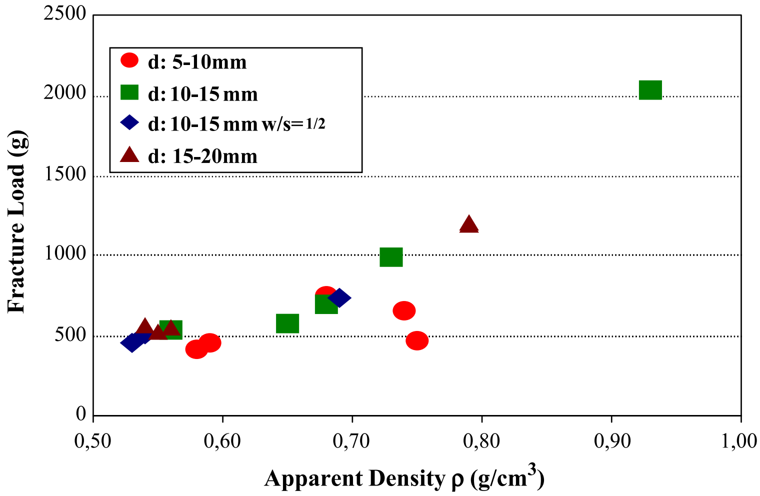

Fig. 7. Relationship of fracture load and apparent density of the LWAs presented in Table 5. *d* = LWA diameter.

section, to measure their fracture load and indirectly their compressive strength. The results are presented in Table 5.

Correlating the fracture loads with the apparent density of each LWA's size and origin group, it is revealed that the fracture load (compressive strength) of the LWAs was proportional to the apparent density, as shown in Fig. 7. Hence, the lightest weight pellets have low fracture loads, being more friable than the denser pellets.

Comparing the diameter of the laboratory-produced LWAs with their sawdust addition and fracture load, it was shown that the LWAs which formed with the highest sawdust addition (5%) have higher fracture load values than the other two, in all size categories (Fig. 8). It is, therefore, assumed that the presence of burnable material in sufficient amounts in the mixture is a key factor for the production of LWAs with good mechanical properties.

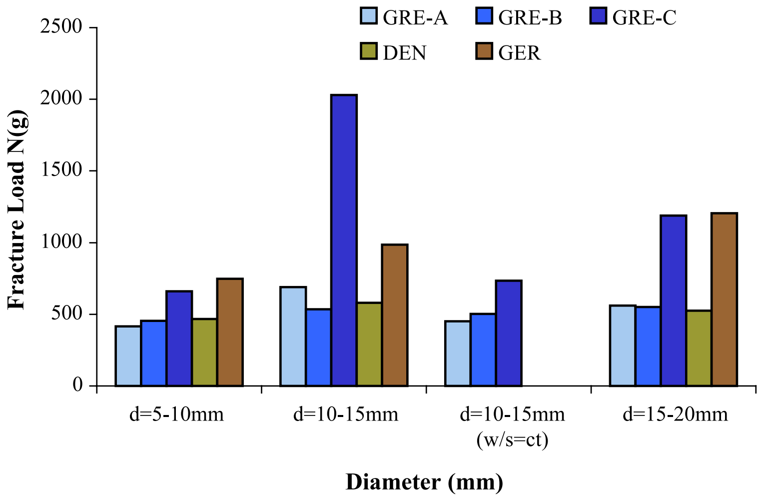

Fig. 8. Strength (fracture load) vs. diameter of the three LWA size categories. GRE-A = Greek LWAs with 2%, GRE-B = 3.5% and GRE-C = 5% sawdust. DEN = Danish LWAs, GER = German LWAs. The third batch of columns refers to the pellets of 10–15 mm in size, produced with constant water/solid ratio (w/s = 1/2).

The 10- to 15-mm-diameter LWAs formed with < 1/2 and 1/2 water/solid ratio have significant differences in their fracture load values, especially in those LWAs formed with 5% sawdust addition (Table 5). It is therefore assumed that the role of the water/solid ratio plays an important role in the manufacturing of LWAs.

5. Conclusions

Fieldwork in Tertiary clayey sedimentary rocks in central Greece resulted in the discovery of a thick sedimentary succession that hosts clayey diatomite deposits [1]. The clayey diatomite rock examined is mainly composed of biogenic amorphous silica (diatom frustules) and expanded clays, such as smectite and vermiculite. Silica, mainly amorphous, alumina and iron are the predominant constituents of the raw materials studied.

The sintered products, either the laboratory or the commercial LWAs, are mostly composed of a glassy phase entrapping honeycomb-shaped voids, ranging in size from 2 to 500 μm. Mullite, hercynite and hematite are among the neoformed minerals present in all three groups of LWAs studied.

The porosity of the LWAs examined, varied. The Greek laboratory-produced LWAs have developed smaller surface porosity than the commercial LWAs. There is a progressive increase of the pore size from the surface inwards in the Greek LWAs. The Danish LWAs are more irregular in shape, having larger pores than the other two, forming a channel system that commonly reaches the surface of the pellets.

In general, the laboratory-produced LWAs have technical characteristics similar to those of commercial origin. The fracture load of the three groups of the LWAs examined is proportional to their apparent density. The Danish LWAs that have the largest pores and the lowest density among the samples studied exhibit the lower fracture load, and indirectly the lowest compressive strength.

The addition of 2%, 3.5% and 5% sawdust in the clayey raw material yielded laboratory LWA products with different technical characteristics, such as apparent density and the fracture load. The best results concerning sufficient strength and low apparent density are obtained when burning mixtures of the clayey diatomite with 5% sawdust. The water/solid ratio plays an important role during manufacturing of the LWAs.

In summary, based on the chemical, physical and mineralogical analyses and tests on the raw materials and the sintered products, it is found that the clayey diatomite originating from central Greece could find applications in the production of LWAs that may be used in the industrial construction sector of the industry, in agriculture applications, such as their use as substrates in hydroponics, in growing media and in greenhouse cultivation in general, in gardening, in civil engineering and other end uses [14–16].

However, the production of LWAs on an industrial scale and for distinct and diverse applications requires further research, as additional parameters involved have to be measured. Among them, raw materials homogeneity, LWAs permeability, water absorption, colouring, aggregate size distribution, pore shape and distribution seem to be the most important ones that could greatly influence the properties and the end uses of the final products [17–20].

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