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Research Article

Effect of different ashes from biomass olive pomace on the mechanical and fire properties of gypsum-based materials

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Abstract: In this study, biomass ashes from different energy valorization processes and storage conditions were used to make fire-resistant materials. Some of the ashes were subjected to a carbonation process. An 80/20 ash/gypsum ratio was used in all compositions. The density and different mechanical properties (compressive and flexural strength, superficial hardness, and dynamic modulus of elasticity), as well as fire resistance properties (insulating capacity and heat absorption capacity), were evaluated at 28 days. The energy valorization had a great influence on the particle size and the Loss On Ignition (LOI) of the fly ash. By increasing both, materials with lower mechanical properties (90%) were produced. Fire resistance was similar for the different ashes tested, but 50% lower than the gypsum material. When the ashes of the materials were carbonated, the material increases compressive strength by 400% compared to ashes without the carbonation process, and the fire resistance was similar to those materials composed exclusively of gypsum, but also a source of CO2 capture is produced.

Keywords: biomass ashes, olive pomace, energy valorization, fire resistance, mechanical requirements.

1. Introduction

Olive pomace, also called orujillo, is the biomass obtained during the extraction of olive oil. Olive pomace is the main solid waste resulting from the production of olive oil. Collectively, 60% are skin and pulp, and 40% are stones (olive husk or pits). Around 0.27 tons of oil and 0.73 tons of olive pomace are obtained per every ton of processed olives, and, therefore, about 3,000,000 tons per year of olive pomace in an average campaign are generated only in Spain, with an approximate humidity between 60-65%.

Typically, in Spain, olive pomace was disposed of in landfills or large tracts of land with poor soil as bio-fertilizer (Valta et al., 2015), but it can produce a possible toxic effect. Nevertheless, new alternatives to disposal are being studied. In Andalucía (South of Spain), many initiatives on the use of olive pomace for energy purposes (dry olive pomace possesses 14,600 kJ/kg as the mean calorific value) are currently undergoing; this biomass is mainly focused on its combustion in boilers or furnaces, in order to generate steam or electric energy. Currently, approximately 30% of the olive pomace generated in Andalusia goes to the production of electricity (Consejería de Agricultura y Pesca – Junta de Andalucía, 2010).

In an average campaign, about 740,000 tons per year of olive pomace are consumed in the 18 Andalusian power plants and another 200,000 tons for self-consumption in the industry itself. There are different systems for burning pomaces, such as rotary kilns, screw burners, and fluidized bed combustion, but the most widely used are grill systems. These can be fixed grills, mobile grills, vibrating grills, and inclined grills. The latter is the most used commercially since they can be adapted to biomass with different particle sizes and water content.

Between 4% and 8% of the burnt-olive pomace is converted into fly and bottom ashes (Consejería de Agricultura y Pesca – Junta de Andalucía, 2010). Due primarily to the high potassium content of the olive pomace ashes, they have been used mainly as fertilizers (Nogales et al., 2011). Other studies show its use as a soil amendment (Nogales et al., 2006), as a low-cost adsorbent (Chan et al., 2017), glasses for semi-conductor applications (Sharma & Singh, 2019), geopolymer-lightweight aggregates (Río Merino et al., 2020; Nguyen et al., 2019; Rashad, 2020), and as a brick component (Moudache et al., 2021; Pérez-Villarejo et al., 2020; Eliche-Quesada & Leite-Costa, 2016; Fernández-Pereira et al., 2011).

Compared to coal combustion ash, biomass shows very peculiar properties, e.g., a high content of alkaline components and unburned matter. These properties invalidate their use in typical recycling applications in large quantities, such as concrete and cement additions (Jidrada et al., 2016), but these characteristics might be useful in fire insulating products (Beh et al., 2021). Consequently, new lines of research should be pursued on the recycling of this type of ash.

Due to the non-inherent resistance to fire of various construction materials, solutions regarding their fire protection are essential to be found (Prager et al., 2020; Vilches, Leiva, Olivares, Vale, & Fernández, 2005). These measures can be classified into two groups: i) active (automatic detection and extinction systems), and ii) passive (slowing down the spread of the fire). Within this last group, the main goal is to keep the temperature of the component below its critical value, as long as possible to avoid its collapse (Wu et al., 2021)].

As commented previously, the ashes of olive oil pomace contain relevant percentages of alkaline elements (potassium and, to a lesser extent, sodium) and alkaline elements of the earth (calcium and magnesium). After combustion, these elements appear in the form of oxides. During cooling, wetting, and subsequent storage, oxides are partially transformed into hydroxides, carbonates, and bicarbonates, changing the nature of ash (López et al., 2018) and transforming it into a product with options to become a fire protection solution (Magnano et al., 2021).

In this work, the physical, mechanical, and fire resistance properties of a construction material composed by 20% wt. of gypsum and 80% wt. of different types of ashes from various combustion processes using olive pomace biomass have been studied. The effect of a previous carbonation process of fly ashes on the different properties of the construction material has also been analyzed.

This product would allow the end-of-life cycle of olive oil production to be closed with a material of high value added. Furthermore, the environmental impact caused by waste disposal in landfills is reduced by recycling this waste in this way.

2. Materials and methods

2.1. *Materials*

2.1.1. Gypsum and olive pomace ashes

In this study, four different olive pomace ashes and one commercial gypsum were properly combined. All of the ashes came from different energy generation processes, which only use olive pomace in an inclined grill oven. O-LOM and E-LOM are fly and bottom ashes from the LA LOMA power plant (Jaén, Spain) with a power of 16 MW. O-ESP is a fly ash from the ESPUNY power plant (Córdoba, Spain) with a power of 17 MW. LO ashes were produced on a laboratory scale (5 hours at 750ºC) in the Escuela Superior de Ingenieros (Seville, Spain) on a fixed grill. Gypsum, according to the literature (CEN EN 13279-1:2009), was used as a binder. The oxide composition of different biomass ashes is listed in Table 1.

Table 1. Major components of different ashes of olive pomace fly.

Parameters/olive pomace fly ashes	O-LOM	E-LOM	O-ESP	LO	Gypsum
Humidity (%)	2.1	1.7	5.3	0.0	6.1
Loss on ignition (% wt.)	9.4	9.3	9.8	0.0	9.1
CaO (% wt.)	17.3	16.2	19.7	15.1	43.7
MgO (% wt.)	6.3	5.0	3.8	6.8	2.0
Fe2O3 (% wt.)	5.5	4.2	1.6	2.1	0.3
Al2O3 (% wt.)	7.0	10.3	0.8	2.6	0.7
SiO2 (% wt.)	36.8	45.4	29.9	19.7	-
K2O (% wt.)	22.5	17.1	22.9	33.1	-
Na2O (% wt.)	1.5	1.7	1.8	0.7	-
SO3 (% wt.)	-	-	-	-	49.9
D50 (μm)	60	80	180	150	27

Regarding the chemical composition, all olive oil pomace ashes have similar main components. These were silica, potassium, and calcium, and they are very similar in all the different types of ashes. Furthermore, the aluminum content is lower than this Si, K, and Ca (Pérez-Villarejo et al., 2020; Eliche-Quesada & Leite-Costa, 2016; De la Casa & Castro, 2014).

As can be seen in Table 1, the main difference between these olive pomaces fly ashes was related to the content of unburned matter (LOI), this being a consequence of the different types of systems used for energy production. The humidity in the olive oil pomace was variable, unlike the coal ash (Alcazar-Ruiz et al., 2021).

From the point of view of particle size, the ashes produced in a furnace without movement (LO) had a larger size than those produced in an industrial grill (Table 1), and the bottom ashes had a larger size than the ashes produced in the same system.

2.1.2. Tested blends

With the aim of establishing a clear comparison of the effect of different types of olive oil pomace ashes in the pastes according to Table 2, a composition of 80% wt. of fly/bottom ash and 20% wt. of gypsum was used for all ashes according to previous studies (Leiva et al., 2005; Leiva et al., 2007). An additional composition with O-LOM ashes was also manufactured after 16 weeks subjected to a carbonation process.

Table 2. Tested blends.

	Type of ash	Ash (% wt.)	Gypsum (% wt.)	Water/solid ratio
B1	O-LOM	80	20	0.4
B1-C	O-LOM carbonated	80	20	0.4
B2	E-LOM	80	20	0.4
B3	O-ESP	80	20	0.4
B4	LO	80	20	0.4
100% Gypsum	-	-	100	0.4

As can be seen in Figure 1, each paste was prepared by mixing the ash and gypsum for 3 min. The water/solids ratio of all mixes was kept constant with a value of 0.4 and mixed again for another 3 min. Subsequently, the pastes were poured into the mold, unmolded after 24 hours, and cured at an average temperature equal to 20ºC and an average humidity of 50% for 27 days more.

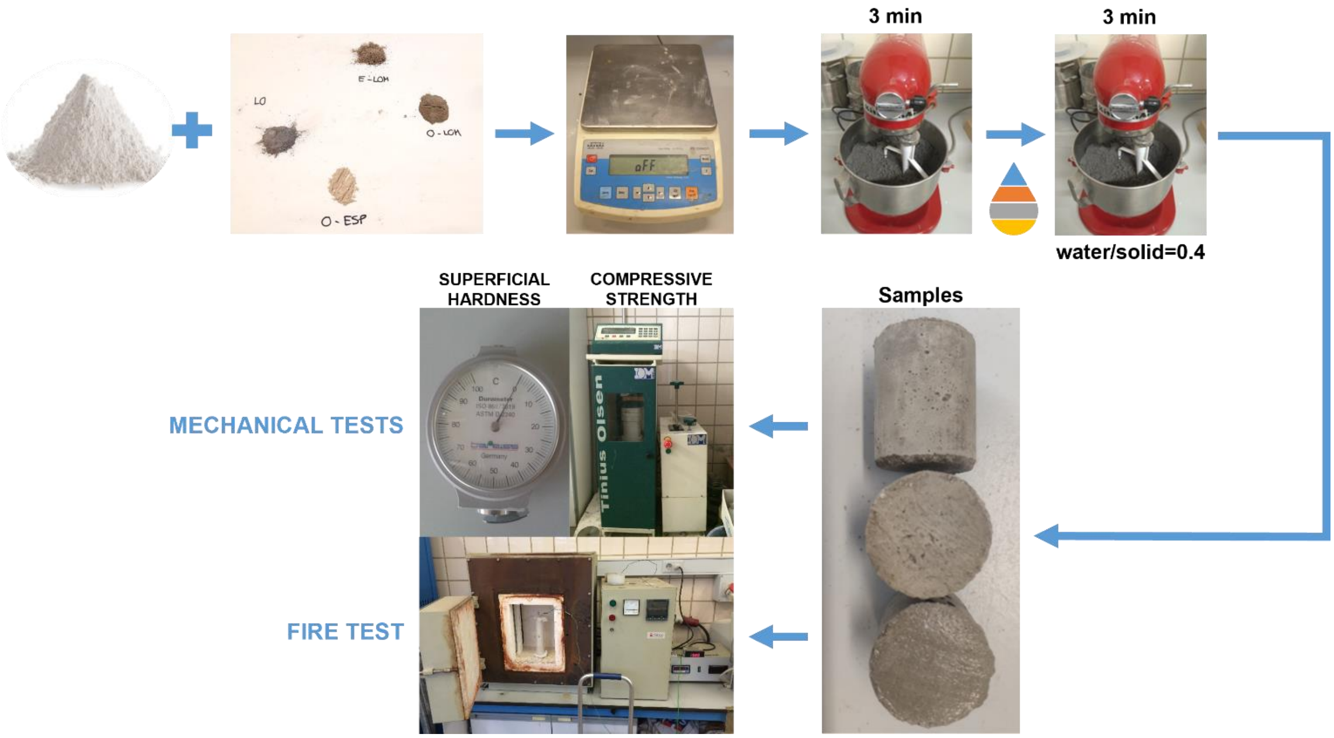

Figure 1. Sample preparation and testing.

2.2. *Test methods*

2.2.1. Density and mechanical properties

The density of the biomass pastes was determined by their weight and volume. For each composition, five specimens were measured.

The compressive strength was determined according to the standard (CEN EN 12859:2012). Four prismatic samples of 16 x 4 x 4 cm for each composition were tested using a compression machine (Suzpecar, MEM-102/500kN).

Surface hardness was determined according to the standard (CEN EN 13279-2:2014). The method is based on the resistance to penetration of a Shore C durometer obtained by a panel. This test was executed twice on 2 cm thick panels. The surface area was 16 cm^2.

The ultrasonic method (CNS Electronics Ltd, CNS electronic portable ultrasonic non-destructive tester) was used to estimate the panels' dynamic modulus of elasticity (E_D); the velocity of the ultrasonic wave is proof to be related to the E_D according to the equation:

$$E_D = K \cdot v^2 \cdot \rho \tag{1}$$

where K is a constant that is related to the Poisson coefficient of the material, v is the velocity of the ultrasonic pulse propagation, and ρ is the density of the paste (Hernández-Olivares & Barluenga, 2004). The tests were carried out on 33-mm-diameter and 40-mm-height cylinders.

2.2.2. Thermal properties

The criterion used to measure thermal insulating capacity was similar to the actual conditions used to test construction materials such as steel sections for passive protection (CEN EN 1363-1:2021). Figure 2 shows a diagram of the test setup to determine the insulating capacity of a sample. Cylinders with dimensions of 50 x 200 mm (diameter x height) had to be placed in an oven and subjected to an external temperature according to the existing literature (Leiva et al., 2005; CEN EN 1363-1:2021). The temperature of fire should rise according to the law:

$$T_{fire} = 20 + 345 \cdot \log(t+1) \qquad (2)$$

where T_{fire} is the fire temperature (ºC), and t is the time (minutes). As indicated in the European standard, the determination of the insulating capacity is defined as the necessary time to reach 600ºC (t_{600}) in the center of the cylinder (T_{in}). The cylindrical assemblies were insulated on the bottom and top surfaces using ceramic fibers with very low thermal conductivity. Subsequently, only a radial and symmetric heat flow is produced during the test.

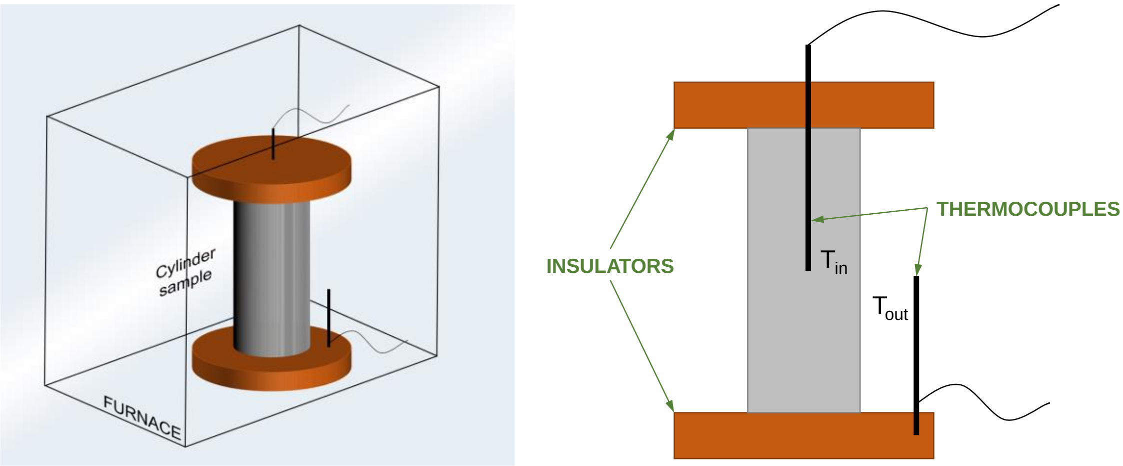

Figure 2. Schedule for the fire insulating capacity test.

The Differential Scanning Calorimetry (DSC) test was used, in order to measure the energy absorbed by the pastes during the heating. The samples were placed in aluminum containers and subjected to heating of 3ºC/min from 30ºC to 300ºC, nitrogen was used as purging gas (Vilches, Leiva, Vale & Fernández-Pereira, 2005). To identify the weight ratio of the different endothermic peaks in the pastes, a thermogravimetric study (TG-SDTA Mettler Toledo 851) was carried out. A heating rate of 10°C/min was chosen, using air as the purging gas.

3. Results and discussion

3.1. *Carbonation process*

A certain evolution in the ashes was observed over time due to its carbonation test, produced by its high potassium content, which also produced high water retention (Cheng & Chiu, 2003) and environmental conditions during its 16-week storage (temperature = 20-25ºC; humidity = 50-60%, 1-3% CO_2) (Vassilev & Vassileva, 2020; Ohenoja et al., 2020; Supancic et al., 2014; Suescum-Morales et al., 2021).

The simultaneous action of both circumstances facilitated the carbonation of orujillo ashes (O-LOM). This produced the progressive formation of para-alumohydrocalcite ($CaAl_2(CO_3)_2(OH)_4 \cdot 6H_2O$ and kalinicinite ($KHCO_3$). Figure 3 shows the diffractograms of the ashes after combustion (non-carbonated) and after 16 weeks (carbonated). Furthermore, these ashes had a high potassium content in the forms of sylvite (KCl) and arcanite (K_2SO_4).

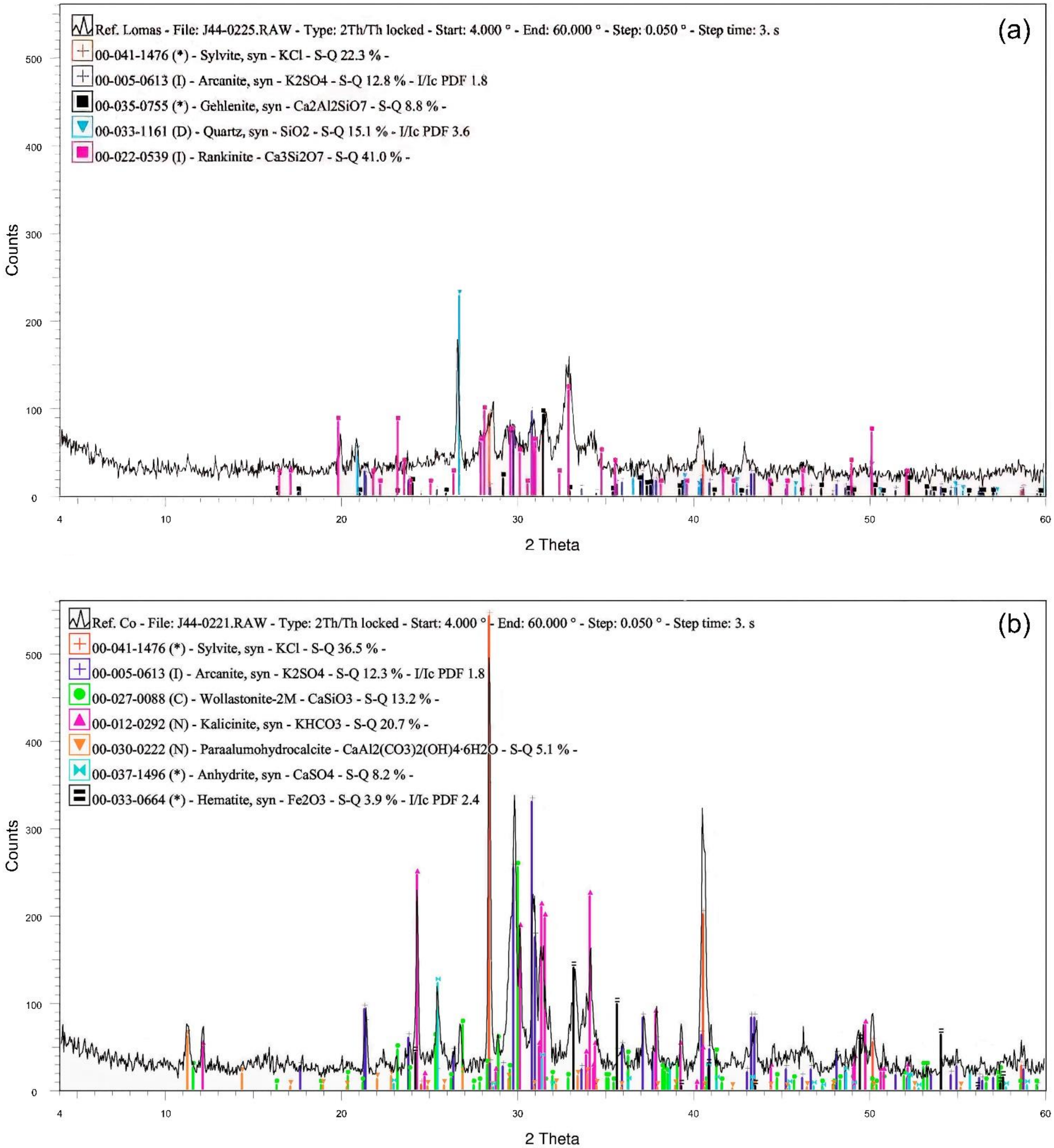

Figure 3. X-ray diffractograms: (a) non-carbonated, and (b) carbonated fly ashes.

When these ashes were subjected to the DSC test, the compounds produced during carbonation caused the appearance of new endothermic peaks between 100 and 200ºC, due to the release of the water contained in the para-alumohydrocalcite (Jitianu et al., 2000), as shown in Figure 4, significantly increasing the energy absorbed by the ash during the heating process.

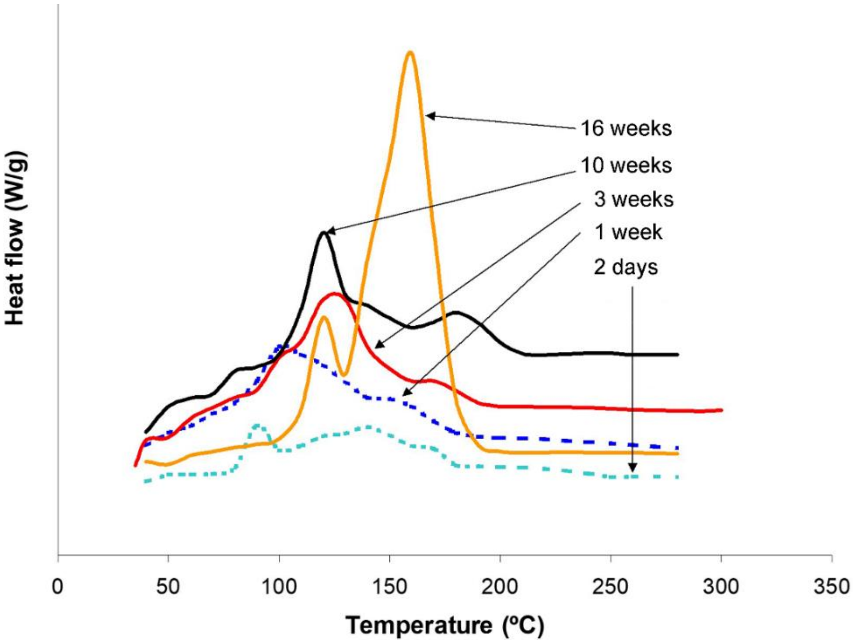

Figure 4. Release of water in O-LOM as a result of the carbonation process.

As can be seen in Figure 4, two days after its production, the ashes showed only a small peak at 90ºC in the DSC test. The amount of chemically bonded water increased during the carbonation process. The first water molecules were released at 90ºC (Stachowicz et al., 2015). When the chemical bonded water was increased, a new peak appeared at 128ºC. This displacement corresponded to a of "self-agglomeration" (Figure 5). The dehydration process of $CaAl_2(CO_3)_2(OH)_4 \cdot 6H_2O$ process of the last water molecules was produced at 150ºC, because they were bounded in a stronger way (Stachowiczn et al., 2015).

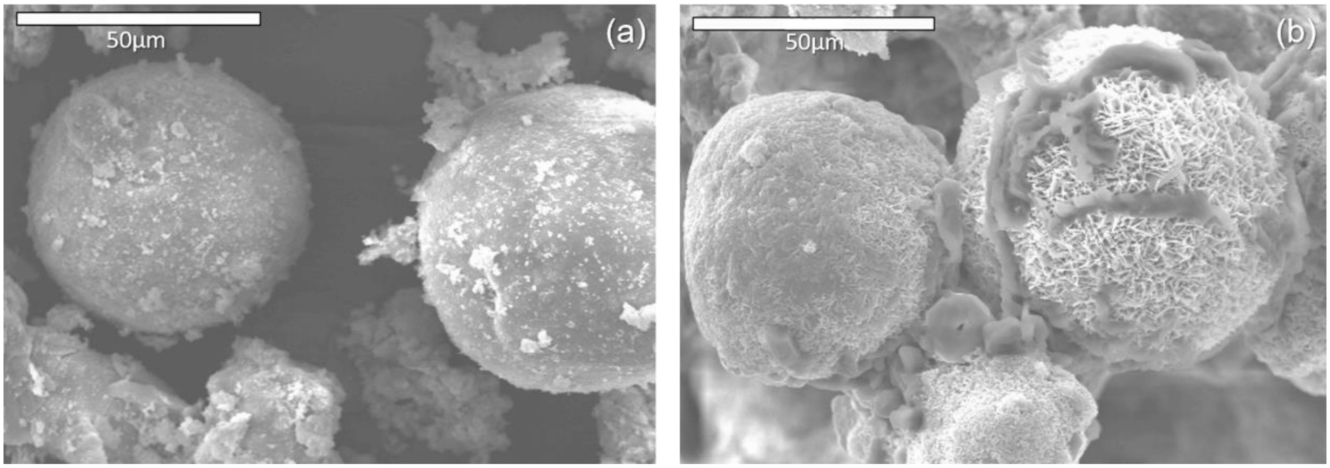

Figure 5. SEM-images from the ashes: (a) non-carbonated, and (b) carbonated.

3.2. *Physical and mechanical properties*

Table 3 shows the density (ρ), compressive strength (R_C), dynamic modulus of elasticity (E_D), and superficial hardness (H) of pastes with different types of olive oil pomace ash.

Table 3. Physical and mechanical properties.

	80% wt. ash - 20% wt. gypsum					
Parameter	B1 O-LOM	B1-C O-LOM (16 weeks)	B2 E-LOM	B3 O-ESP	B4 LO	100% Gypsum
ρ (g/m3)	1078	1270	1028	894	910	1371
RC (MPa)	0.9	4.2	0.7	0.3	0.5	8.3
H (Shore C)	44	72	22	30	21	90
ED (GPa)	1.4	2.9	0.9	0.5	0.4	8.1

As can be seen, the density of the pastes depended on several factors. The first was that each one had a different particle size; the larger the particle size, the higher the porosity produced, and, therefore, the lower the density. Second, the density also depended on the specific gravity of the particles, which depended on the unburned content due to its very low specific gravity compared to the ash itself (Leiva et al., 2008). Regarding the LOI, it depended largely on the type of process or grill used. When the LOI was high, internal porosity also increased, leading to a reduction in density, and therefore, lower mechanical properties (Leiva et al., 2008). Finally, it also depended on the degree of carbonation of the ashes. Carbonation decreased the volume of pores between the particles (Figure 5), thus increasing the density of the materials.

The compressive strength was inversely proportional to the porosity of the material and, hence, its density; therefore, it was affected by the same factors. Carbonation significantly improved all mechanical properties (R_C, H, and E_D). When comparing pastes with low carbonated ashes (O-LOMAS) with more carbonated ones (O-LOMAS after 16 weeks), an increase in mechanical properties was observed.

The dynamic modulus of elasticity varied significantly with the type of ash. Those with higher densities produced pastes with higher E_D and, predictably, had a higher flexural strength (Leiva et al., 2018).

3.3. *Insulating capacity*

The average temperatures in the center of the cylinders during the fire test are shown in Figure 6.

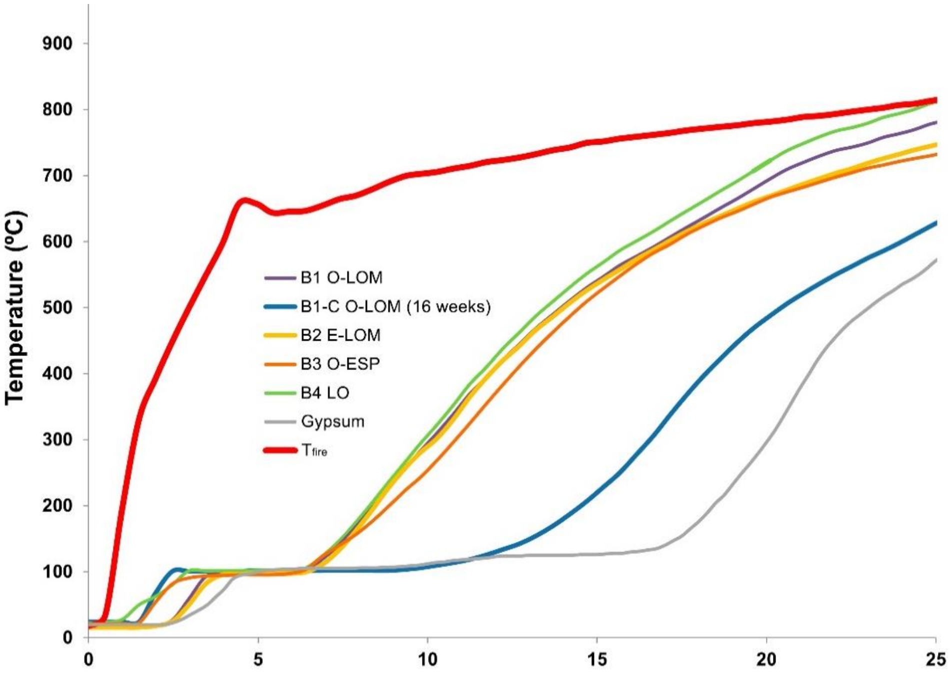

Figure 6. Evolution of the temperature in the center of the cylinder versus the exposure time to a standard fire.

The porous material can contain free, adsorbed, and/or crystallized water. When this water evaporates as a result of a heating process, such as a fire, it produces an overpressure inside the paste. Due to such an overpressure, this steam is routed to inner zones in the cylinder; as these areas are colder, the water steam cools down and, subsequently, condenses again. As a result, a liquid film is created, that is slightly displaced and moved to the core of the cylinder. While this is happening, the fire energy is consumed in this process, maintaining the temperature around 100ºC in the center of the material. This effect leads to the appearance of an evaporation plateau, a period in which the temperature in the center of the cylinder is constant, as shown in Figure 6 (Ríos et al., 2020).

Figure 6 shows that pastes with different fly and bottom ashes from orujillo presented a similar insulating capacity (t_{600}), but smaller than that obtained by a material composed solely of commercial gypsum. According to Figure 7, a first endothermic peak is produced between 100ºC and 120ºC, due to moisture evaporation and chemically bonded water from gypsum. The gypsum was transformed into calcium sulphate releasing the water ($CaSO_4 \cdot 2H_2O \rightarrow CaSO_4 + 2H_2O$) (Leiva et al., 2010). As all pastes were placed under the same moisture conditions and contain the same gypsum content, this peak is similar for all compositions with orujillo. By containing only 20% gypsum, the pastes with the different types of fly and bottom ashes reduced the duration of their plateau and, therefore, the t_{600} compared to 100% gypsum.

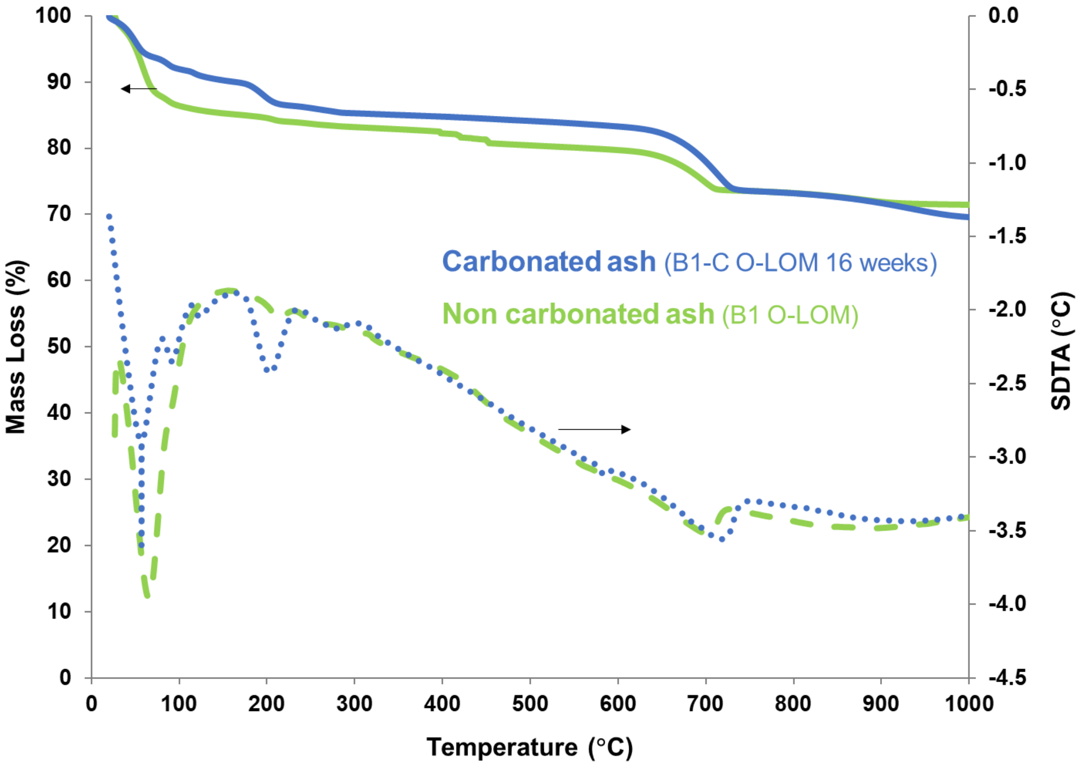

Figure 7. Thermogravimetric study of carbonated and non-carbonated orujillo pastes.

For pastes with carbonated ash, t_{600} increased markedly. The temporal duration of the plateau increased due to the appearance of new compounds that were endothermically decomposing (Figures 4 and 7), increasing the energy absorbed during heating and making the t_{600} quite similar to the paste with only commercial gypsum. The weight loss of carbonated orujillo paste from 200°C to 650°C is due to the evaporation of other adsorbed and crystalline water and the water decomposition of hydroxides, according to the literature related to other similar materials (Jin et al., 2000; Asako et al., 2004).

Other fire resistance materials containing other wastes, such as fly and bottom ashes (Vilches, Leiva, Vale, & Fernández-Pereira, 2005), slag (Ríos et al., 2020), or geopolymers (Galiano et al., 2017), show a lower thermal insulating capacity than fly and bottom ash materials under the same test conditions.

During the fire tests, no gas emissions were observed. Moreover, the cylinders did not show deformations during the whole test (before, during, and after).

4. Conclusions

In this work, the influence of different ashes from biomass olive pomace of different energy processes mixed with gypsum, in a ratio of ash/gypsum equal to 4, on the physical, mechanical, and fire resistance properties has been studied. The conclusions were the following:

1. The recycling of biomass ashes as a fire-resistant product could be an excellent opportunity, instead of storing them in landfills;
2. Energy valorization had a great influence on the particle size (between 60 and 150 µm) and LOI (between 0 and 9.8%) of the biomass fly ashes generated. On the one hand, when the particle size was increased, the density decreased (10%), which worsened the mechanical properties (40%). On the other hand, when the LOI was increased, lower mechanical properties were achieved;

3. The carbonation process increased mechanical properties (400%) and fire resistance was similar to those of commercial products based on gypsum; this is due to the formation of para-alumohydrocalcite, which is endothermically decomposed during fire between 100 and 150°C. Furthermore, this process acted as a CO_2 sink, thus a double environmental objective was achieved: reuse of waste and capture of CO_2.

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