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Enhanced Thermo-Physical Properties of Gypsum Composites Using Olive Pomace Waste Reinforcement

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ABSTRACT

Today, the construction sector consumes 30-40% of the world's total energy and contributes one-third of total greenhouse gas emissions. Consequently, the development of new eco-friendly building materials with improved properties is becoming increasingly important. Olive pomace waste is released into the environment, which has a negative impact on it. Recycling this olive pomace waste as an alternative raw material in the construction industry can protect the environment and at the same time reduce the additional costs of managing and disposing of this waste for local authorities. It is an environmentally friendly and sustainable solution to waste recycling. This study investigated the effect of adding olive pomace (OP) to building materials. Four proportions of this additive (4%, 8%, 12% and 16%) were used. Physical, thermal properties (conductivity and diffusivity) as well as mechanical properties (compressive and flexural strength) of the composites were carried out. The traditional gypsum-based composites had a thermal conductivity of 0.478 $W.m^{-1}.K^{-1}$, while the composites of gypsum with additive show an interesting thermal conductivity of 0.390 $W.m^{-1}.K^{-1}$ for a percentage of 16% (OP16) with a reduction rate of 22.56%, and mechanical properties lower than those of the reference gypsum-based composite but in accordance with the standard EN 133279-1, with compressive strength of almost 4.10MPa for a percentage of 16% (OP16), and flexural strength equal to 2MPa. This is due to the increase in porosity as indicated by the microstructure of the composites. We also tested water absorption by capillary action for each specimen, and found that this coefficient increased with increasing percentage of waste.

1. INTRODUCTION

In the new energy framework, biomass plays a very important role, given the relatively large quantity of residues produced worldwide. Olive groves have two main uses: the production of table olives and olive oil. In Morocco, olive production for the 2021-2022 season is estimated at 1.96 million tonnes, up 21% on the previous season. Average annual exports of table olives are 91,300 tonnes/year, 15,000 tonnes/year of olive oil and 13,700 tonnes/year of olive-pomace oil [1]. This puts Morocco in third place for table olive exports and ninth place for olive oil exports. The main waste product generated is olive stones. In fact, olive pomace is one of the most abundant materials in Morocco, but it is less valued and poorly considered. Olive pomace can be used in animal feed [2, 3], as fuel [4, 5], or to fertilise agricultural land [6].

This waste can also be used in the production of building bricks to improve their thermo-physical properties. Olive pomace waste is discharged randomly into the environment, creating a negative environmental impact. Recycling this olive pomace waste as an additive in the construction industry not only reduces industrial waste but can also minimise the additional cost imposed on the state for its disposal. At the same time, it is an environmentally friendly and sustainable way of recycling this type of waste. Several advantages of incorporating plant aggregates have been cited in the literature in order to develop new materials with different use criteria. With regard to thermal behaviour, low thermal conductivity is a criterion that makes it possible to improve energy consumption. Several researchers have used olive kernel waste to manufacture a composite, either clay-based [7-9] or using cement mortar [10], while others use self-compacting concrete [11].

Lila et al. [12], used olive pomace as an additive in cement mortar to monitor the effect of incorporating this waste product on the mortar's mechanical properties. They measured the compression resistance at different ages, they found that the compressive strength of the additive mortar decreased significantly compared with the reference mortar after 28 days, and became negligible after 365 days, whereas the compressive properties of the mortar containing 10% of the additive were very close to those of the reference cement mortar.

El Hammouti et al. [8] studied the effect of incorporating olive pomace into a composite based on local soils. They found that the thermal conductivity of the elaborated bricks (fired and dried) decreased with the addition of OP. The thermal diffusivity of the dried composites with 20% OP decreased by 45.14% and stayed essentially constant for all composites after firing.

Moreno-Maroto et al. [13] developed lightweight aggregates in combination with clays, using olive pomace waste as an additive by varying the additive percentages from 0% to 10%. They found that increasing the additive rate increased the porosity of the clays, and that recycling olive pomace produced environmentally friendly and economical materials. The use of lightweight aggregates offers environmental advantages due to their light weight and high porosity.

Muñoz et al. [14] used olive pomace as an additive to reduce the thermal conductivity of fired clay using the following percentages: 0%, 5%, 11%, 17%. They found that adding 5% olive pomace to the clay reduced thermal conductivity by up to 10%, which meant an improvement in the thermal conductivity of the masonry. The apparent density was reduced linearly to 23% by the addition of 17% additive. The result is a reduction in the load on buildings due to the weight of the envelopes themselves, and consequently a decrease in the associated logistical and manufacturing costs.

Boukhari et al. [15] studied the effect of partial substituting olive waste for sand on thermal and mechanical properties of sustainable concrete, they used substitution percentages from 0 to 15%, mechanical results indicated that composites containing 5% olive waste showed better results, and the thermal conductivity of the composites was improved by reducing it from 1.3 $W.m^{-1}.K^{-1}$ for the control concrete to 0.86 $W.m^{-1}.K^{-1}$ for 15% of replacement.

Kaya et al. [16] studied the filling properties of olive waste powder for the polypropylene matrix material using different percentages between 10 and 40% with a step of 10, they noticed that when 40% by weight of olive waste powder filler was filled into the polypropylene, the Young's modulus and flexural strength of the polypropylene increased by about 62.5% and 19%, respectively, and the thermal properties also improved with increasing powder fraction.

Adazabra et al. [17] used solid olive waste as material to form pores in fired clay bricks. They noted an increase in porosity with the addition of olive pomace waste, and a decrease in the density of the samples. The compressive strength of the bricks fell from 20.01 MPa to 6.3 MPa, but this was offset by a more than 30% improvement in thermal insulation, with thermal conductivity falling from 0.84 $W.m^{-1}.K^{-1}$ to 0.54 $W.m^{-1}.K^{-1}$.

From this review we can see that, on the one hand, using of olive pomace as an additive reduces the compressive strength, but with 10% additives, it gives better results. On the other hand, the thermal conductivity decreases with the addition of olive pomace waste and the density decreases linearly. It was also found that the addition of this waste gave a very porous structure to the composites, which improved the thermal results.

Gypsum has been known as a binder with good technical properties which has led to its widespread use [18]. It is very important to the construction industry because of its low density, its ability to control humidity and heat, its excellent performance in protection against fire, and its low cost [19]. The gypsum is widely used as a bonding material, interior partitions, ceiling tiles and interior wall cladding [20]. As this waste has not been treated with gypsum in the literature, this work assesses the effect of using olive pomace waste on the various physical, mechanical and thermal properties of the gypsum matrix. We used the different mass percentages (0%, 4%, 8%, 12% and 16%) to study their effects on the properties of the gypsum. The mechanical performance of the specimens was verified by compression and flexural strength tests, the thermal characteristics were measured using the hot disc method, and the water characteristics were treated using the capillary water absorption method.

2. MATERIALS AND METHODS

2.1 Raw material

2.1.1 Gypsum

Gypsum is a powder derived from gypsum fired at a high temperature of 150°C according to Eq. (1), used in construction when transformed into a paste. It is a material used as wall and roof cladding. It is known the world over for several qualities, notably its malleability and low density [21].

$$2CaSO_4\,4H_2O + Heat \rightarrow 2CaSO_4H_2O + 3H_2O \quad (1)$$

2.1.2 Olive pomace

In our study, the filler used was raw olive pomace. It was collected from a modern oil mill located in the Sous Massa region. The olive pomace was placed in tubs and washed with warm water to separate it from the margin and olive pulp using a sieve, after washing, the pomace underwent natural air drying for two days to eliminate any moisture that might alter its characteristics, then dried in an oven at 105°C for 24 hours. The clean pomace is crushed using a laboratory grinder, followed by sieving using a sieve with a size of 80μm, this process is shown in Figure 1.

Figure 1. Waste preparation steps

2.2 Characterization of materials

2.2.1 Physical and chemical characterization

By analysing the chemical composition of OP under energy dispersive X-ray spectroscopy (EDS) as shown in Figure 2, we see that it contains 50.32% carbon and 49.68% oxygen. Table 1 presents the physical properties of gypsum and OP, showing that the density of olive powder is lower than that of gypsum, and the porosity of OP powder is higher than that of gypsum.

Figure 3 shows the microstructure of olive pomace powder using scanning electron microscope analysis with the following conditions: a voltage of 10KV, magnification of ×160 with a scale of 100µm and ×3,000 for the scale of 5µm, as shown in Figures 3 and 4. We can see that the olive grain powder has a variable size distribution and a non-smooth surface, we observe the presence of virgin fibres with several pores and also voids, this observation is reported by other authors [22]. On the other hand, we can see from Figure 4 that the morphology of gypsum powder is homogeneous and appears rugose with micropores.

Figure 5 shows the XRD image of olive waste using the Bruker D8 Advnce Twin diffractometer, fitted with a copper anti-cathode type diffractometer (λCu = 1.5418 Å). The diffraction spectrum is scanned between 5° and 80° with a step size of 0.02°. The specimens are ground with a grinder until they become a very fine powder for analysis. The phases identified are Quartz with a percentage of 26%, pyrazole and magnesium dihydrogen phosphate with percentages of 35% and 29% respectively, these phases have been identified by other researchers [23].

Table 1. Physical and chemical properties of the materials

Parameter	Unite	Results	
		Gypsum	Olive Pomace Powder
Water content	%	4.05	3.62
Real density	$Kg.m^{-3}$	1633.33	1583.33
Apparent density	$Kg.m^{-3}$	777.99	505.09
Porosity	%	52.37	68.09

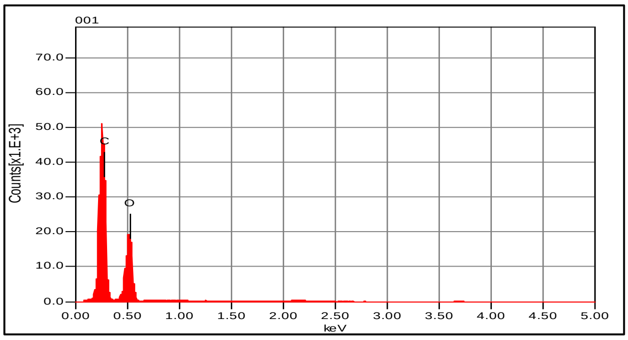

Figure 2. Chemical composition of the OP powder

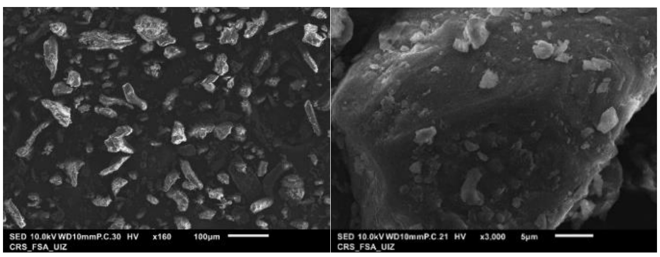

Figure 3. Microstructure of olive pomace powder

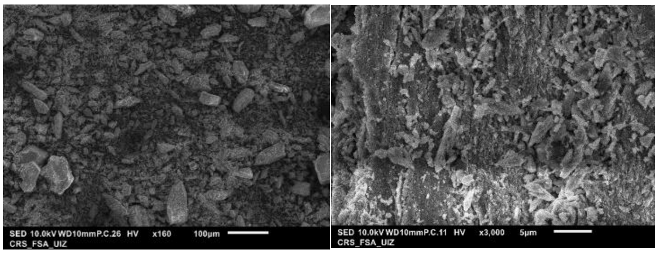

Figure 4. Microstructure of gypsum

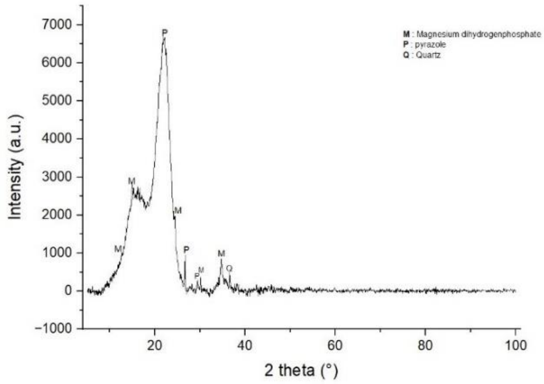

Figure 5. Diffractogram of olive pomace waste

2.3 Composite preparation

After preparing the waste it passed through a sieve smaller than 80 µm it was mixed with gypsum as an additive at different mass fractions (0%, 4%, 8%, 12%, 16%) these capsules were made manually in a rectangular mould (4×4×16 cm^3) for each type, as shown in Figure 6. A proportion of water calculated by the ratio Solid/Water = 1.33 was gradually introduced into the mixture while maintaining manual agitation short enough to avoid hardening, the quantities measured are given in Table 2.

The composite plates were air-dried for 5 hours and removed from the moulds. The samples were conserved for 28 days under laboratory conditions with a temperature of 21°C and relative humidity of 65%.

Table 2. Proportion of materials for the preparation of composites

Percentage of the Additive	Gypsum (g)	Olive Pomace Powder (g)	Water (g)
OP0	270	0	203
OP4	270	10.8	211.1
OP8	270	21.6	219.25
OP12	270	32.4	227.37
OP16	270	43.2	235.49

Figure 6. The composites elaborated

2.4 Methods

2.4.1Thermal properties

The determination of the thermal conductivity of building materials is generally based on several methods. In this study, the thermal properties were determined using a "HOT DISK TPS 1500" device (Figure 7), which can be used to determine thermal conductivity and diffusivity.

The TPS method allows power to be supplied to the Hot Disk probe for a limited time, to raise the temperature of the material under study. The rise in temperature is linked to the variation in the probe's electrical resistance. The characteristics of the temperature as a function of the electrical resistance are recorded, and analysis of this variation is used to determine both conductivity and thermal diffusivity [24]. In our study, we measured the thermal conductivity after 28 days of hardening under laboratory conditions with a temperature of 21°C and relative humidity of 65% in the wet state after mass stabilization.

Figure 7. Thermal properties measurement

2.4.2 Mechanical properties

The mechanical properties were tested using an "AUTOMAX 5" machine shown in Figure 8. These machines, equipped with a very rigid four-column frame, were designed to test the prisms either in compression or in 3-point bending. these tests are carried out in accordance with the standard NF EN 196-1. For the flexural test, we used 4×4×16 cm^3 specimens, which were placed on two supports, and the load was applied vertically on the lateral face, while regularly increasing the load at a speed of (50 ± 10) N.s^{-1}, until failure. The flexural strength R_f is calculated according to the formula bellow in MPa:

$$R_f = \frac{1.5 \times Ff \times l}{b^3} \quad (2)$$

Or,

b is the square cross-section of the prism, in millimetres.

F_f is the force applied to the middle of the prism at the moment of rupture, in Newtons.

l is the distance in millimetres between the supports.

For the compressive test we used 4×4×8 cm^3 prisms, the load is applied to the lateral face of the specimen while increasing the load regularly at a speed of (2,400 ± 200) N.s^{-1}, until failure. The compressive strength R_c is calculated according to the following formula:

$$R_C = \frac{F_C}{1600} \quad (3)$$

Or,

F_c is the maximum breaking load, in Newton's.

1600 is the area of the trays or auxiliary plates (4cm×4cm), in square millimetres.

Wet densities are taken after 28 days of hardening, and are noted once the masses have stabilized.

Figure 8. Compression and flexural test

2.4.3 Water absorption by capillarity

Absorption by capillarity is a hydric property that reflects the stone's capacity to absorb a quantity of water per unit of time and surface, when only one face is in direct contact with water. The water absorption coefficient by capillary C is expressed in Kg.m^{-2}.min$^{-1/2}$, according to norm EN 1015-18 [25].

This test consists of following the kinetics of capillary imbibition through the quantity of water absorbed per unit surface area of the sample in contact with the water, as a function of the square root of time.

The samples used were broken into two pieces measuring 4×4×8 cm^3, dried at a temperature of 70°C until a constant mass was obtained, then weighed to determine the dry mass. The samples were then placed in a tray in which one side of the sample was constantly in contact with between 5 and 10 mm of water as shown in Figure 9. The samples are weighed over time, at two instants, 10 min and 90 min, according to the standard. They are then calculated using the following equation.

$$C = 0.1(M_2 - M_1) \quad (4)$$

where, C is the water absorption coefficient by capillary, in Kg.m^{-2}.min$^{-1/2}$, M_1 and M_2 are the masses of the test piece after immersion for a time t_1 = 10 min and t_2 = 90 min respectively, in g.

Figure 9. Water absorption by capillarity

3. RESULTS AND DISCUSSION

3.1 Physical properties and microstructure of composites

Table 3 shows the wet densities of the materials produced after 28 days of curing in the open air. We see that the density of the composite decreases as the percentage of additive increases with a percentage of reduction for the 16% additive case is 8.80%, since the density of the olive grain powder is lower than that of the gypsum, other authors have noted that the addition of lightweight additives makes it possible to reduce the composite density [26]. The decrease in density implies an increase in the number of pores within the volume due to the evaporation of the water physically bound to the gypsum. This decrease is linked to the thermal and mechanical properties, as we will see in the following sections.

Figure 10 shows the morphology of the composites produced with the different percentages (0%, 8%, 16%). We can see from Figure 10a) that the morphology of OP0 is homogeneous and appeared rugose. But when the OP powder was incorporated, the surface of the matrix was modified, as shown in Figure 10b). The filaments were covered by the new OP coating, creating holes and voids. With the increase in the level of OP powder, we note a complete change in the morphology of the gypsum matrix as illustrated in Figure 10c). we can see that as OP is added to the matrix, the material becomes more porous, this observation has been mentioned by other authors [13, 17]. They are uniformly distributed over the entire surface of the composite and are not agglomerated in a single area. The porosity observed in composites incorporated with olive powder can directly affect thermal properties in a positive way, however, it can be a weak point for mechanical stresses.

Table 3. Density of the different composites

	Density $kg.m^{-3}$				
	ρ_1	ρ_2	ρ_3	ρ_{ave}	$\Delta\rho/\rho$ (%)
OP0	1037.34	1043.50	1089.45	1056.76	3.09
OP4	986.97	1009.08	1036.96	1011.00	2.57
OP8	987.88	988.11	985.80	987.26	0.09
OP12	980.53	974.33	962.77	972.54	0.82
OP16	964.18	936.60	990.62	963.80	2.78

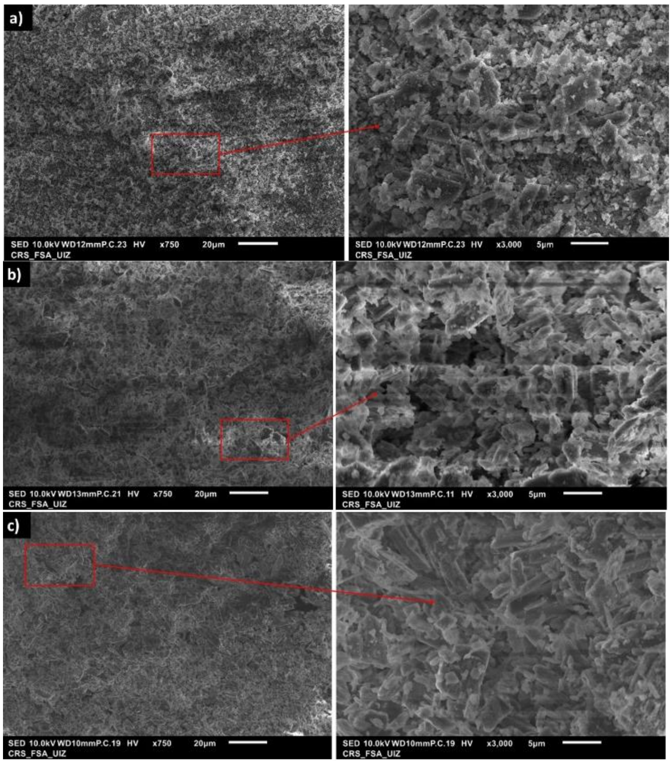

Figure 10. SEM images of the composites a)-OP0, b)-OP8, c)-OP16

3.2 Thermal properties

Table 4 shows the thermal properties of the specimens studied under normal laboratory conditions. We calculated the uncertainty from the following relationships [27]:

$$\frac{\Delta\lambda}{\lambda} = \frac{|\lambda_{max} - \lambda_{ave}|}{\lambda_{ave}} \quad (5)$$

$$\frac{\Delta\alpha}{\alpha} = \frac{|\alpha_{max} - \alpha_{ave}|}{\alpha_{ave}} \quad (6)$$

Table 4. Thermal properties of the materials developed

	Thermal Conductivity ($W.m^{-1}.K^{-1}$)					Thermal Diffusivity ($mm^2 s^{-1}$)				
	λ_1	λ_2	λ_3	λ_{ave}	$\Delta\lambda/\lambda$ (%)	α_1	α_2	α_3	α_{ave}	$\Delta\alpha/\alpha$ (%)
OP0	0.473	0.480	0.481	0.478	0.63	0.408	0.425	0.415	0.416	2.16
OP4	0.420	0.421	0.421	0.421	0	0.432	0.423	0.423	0.433	2.31
OP8	0.419	0.420	0.421	0.420	0.24	0.379	0.387	0.393	0.386	1.81
OP12	0.402	0.403	0.408	0.404	0.99	0.309	0.319	0.329	0.319	3.13
OP16	0.382	0.392	0.397	0.390	1.79	0.291	0.309	0.319	0.306	4.25

Table 5. Flexural and compression test results

	Flexural Strength (MPa)					Compressive Strength (MPa)				
	R_{F1}	R_{F2}	R_{F3}	R_{Fave}	ΔR_F	R_{C1}	R_{C2}	R_{C3}	R_{Cave}	ΔR_C
OP0	3.45	4.06	3.51	3.67	0.61	9.92	10.12	9.78	9.94	0.34
OP4	2.18	2.63	2.90	2.57	0.72	7.59	7.75	6.50	7.28	1.25
OP8	2.53	2.43	3.19	2.72	0.76	6.36	6.76	6.76	6.63	0.40
OP12	2.21	2.94	2.63	2.59	0.73	5.00	4.81	4.93	4.91	0.19
OP16	2.23	1.89	1.89	2.00	0.34	4.11	4.18	4.02	4.10	0.16

The results shown in the table indicate that the thermal conductivity and thermal diffusivity of specimens containing waste are lower than those of pure gypsum. Figure 11 presents the variation in thermal conductivity of the specimens elaborated as a function of the percentage of olive pomace waste. The thermal conductivity of the specimens varies from 0.478 to 0.390 $W.m^{-1}.K^{-1}$, which represents a reduction of 18.41%, reflecting an improvement in the insulation of the samples processed, so that several authors can deduce that the addition of olive pomace to the matrix has a positive effect on the thermal insulation of composites [8, 14]. This can be explained by the creation of pores inside the composites (Figure 10), which favours the infiltration of air inside the specimen, since air is a good insulator, it has a positive effect on the thermal properties of composites. The Figure 12 shows the variation in thermal conductivity of the various composites as a function of density. It can be seen that thermal conductivity and density vary linearly, so that thermal conductivity decreases as the density of the composites decreases. These results are approved by several researchers who have found that when the density of the composite decreases, porosity increases, which directly implies a reduction in thermal conductivity and consequently an improvement in thermal performance [26-29].

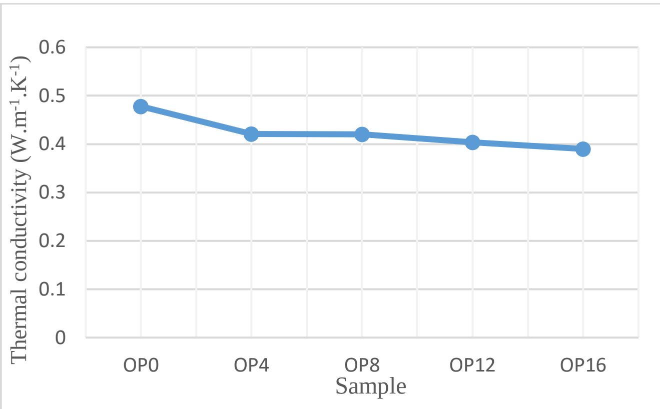

Figure 11. Thermal conductivity as a function of OP percentage

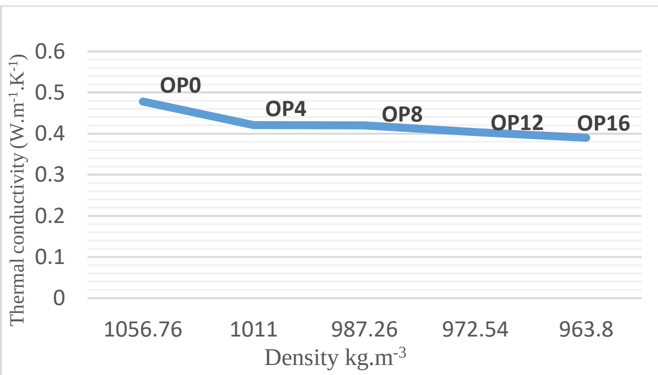

Figure 12. Thermal conductivity as a function of density of different composites

3.3 Mechanical properties

Table 5 indicates the results of mechanical test measured after 28 days. The compressive and flexural strength at three points were measured for three specimens for each percentage and the average value for each was calculated, we measured the uncertainty of each case by the difference between the maximum and the minimum value.

Figures 13 and 14 show the variation in compressive and flexural strength of composites as a function of additive percentages. Compressive and flexural strength are reduced with the addition of the additive this result was obtained by other authors [15, 16]. Compressive strength decreased from 9.94 MPa in the reference case to 4.10 MPa in the case of 16% additive with a percentage of 58.75%, and flexural strength decreased from 3.67 MPa to 2 MPa for 16% additive with a percentage of 45.50%, which shows that the addition of olive pomace waste does not improve the mechanical behaviour of the material, but these results comply with norm EN 133279-1 [30] for all the percentages in the case of compression and flexion since they are greater than 2 MPa and 0.83 respectively. The reduction in mechanical properties means that the application of this type of composite is limited to the production of moulded products (decorative ceiling, smooth false ceiling, cornice, rosette, arches, column, etc.) and for the fixing of these same products.

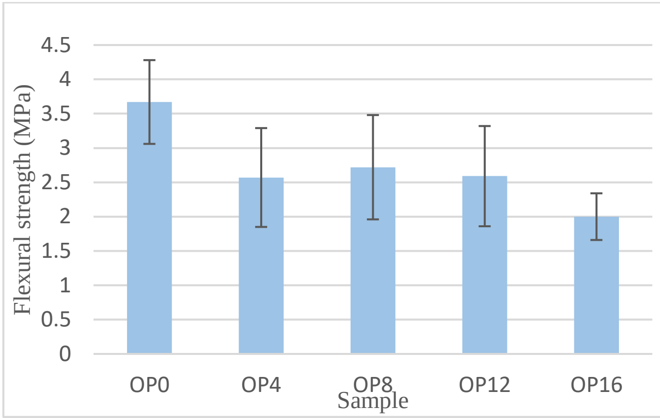

Figure 13. Flexural strength for different samples of mixtures

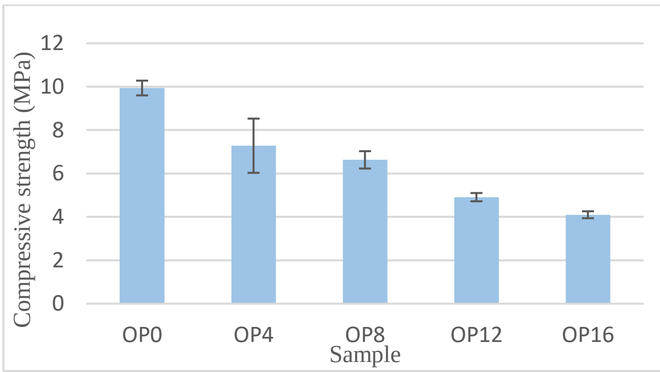

Figure 14. Compressive strength for different samples of mixtures

3.4 Water absorption by capillarity

Figure 15 shows the variation in the water absorption coefficient for each material. We see that the inclusion of this waste increased the coefficient of water absorption by capillary action from 3 $Kg.m^{-2}.min^{-1/2}$ for the reference material OP0, to 4.18 $Kg.m^{-2}.min^{-1/2}$ for OP16, which can be explained by the porosity effect, since adding waste makes the material more porous, so the absorption rate increases. This is mentioned by Khatib [31], which showed that an increase in the total porosity of concrete is strongly associated with an increase in its absorption. These capillary absorption values remain normal compared with those obtained by other researchers, which are greater than 10 $Kg.m^{-2}.min^{-1/2}$ [32]. Taking into account that the rate of increase of 16% of additive is about 39% which remains less than half. High levels of humidity due to capillary action have a number of consequences: blistering of gypsum work, black stains. Undesirable aesthetic effects visible on the interior walls of houses, especially in a damp climate.

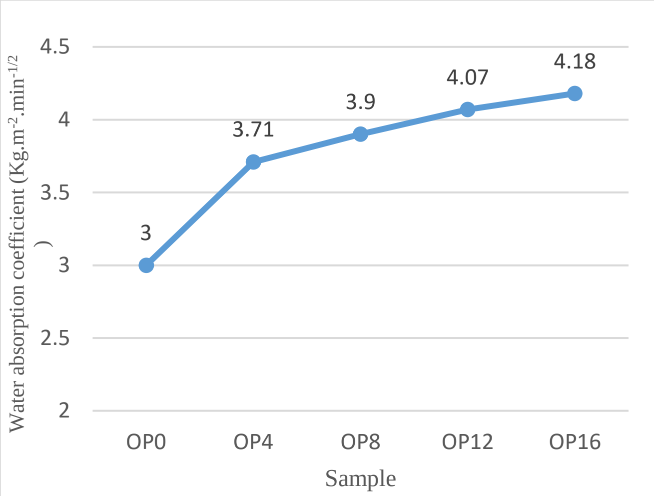

Figure 15. Water absorption coefficient as a function of the percentage of OP in the matrix

4. CONCLUSIONS

This study developed a gypsum-based composite material using olive pomace waste as an additive, with the aim of manufacturing a new ecological material and improving their thermal and mechanical properties. We varied the percentages with a step of 4% by mass, and developed 5 materials (0%, 4%, 8%, 12%, 16%). We studied the physical and chemical characteristics of gypsum and olive pomace waste, and then carried out a mechanical and thermal characterisation of these innovative materials for the different mass percentages used. The rate of water absorption by capillary action was also calculated. We found the following results:

1. According to the results of DRX analysis, the olive grain waste is mainly constituted of Quartz, pyrazole and magnesium dihydrogen phosphate with percentages of 26%, 35% and 29% respectively.
2. Incorporating olive pomace powder into the gypsum matrix creates voids and pores, increasing the porosity of the materials
3. The densities of the specimens produced decreases with the incorporation of olive waste into the composite, the percentage reduction for the 16% additive case is 8.80%,
4. Thermal conductivity was reduced from 0.478 for pure gypsum to 0.390 $W.m^{-1}.K^{-1}$ for 16% of additive, with a percentage reduction of 18.41%, this reduction justified by the increased porosity of the materials elaborated.
5. The addition of waste reduced flexural and compressive strength by 45.50% and 58.75% respectively, with compressive strengths of almost 4.10MPa for a percentage of 16% (OP16), and flexural strength equal to 2MPa, but these results are conformed to the norm EN 133279-1.
6. The addition of waste increases the water absorption coefficient by 39%, from 3 $Kg.m^{-2}.\ min^{-1/2}$ to 4.18 $Kg.m^{-2}.\ min^{-1/2}$ for OP16%, explained by the effect of porosity on the water absorption rate.

In conclusion, adding olive pomace waste to the gypsum matrix produces a new environmentally-friendly material with improved thermal conductivity, which decreases with the addition of this waste, with a gain of 18.41% for OP16. The composite developed in this study can be used for the production of moulded products (decorative ceiling, smooth false ceiling, cornice, rosette, arches, column, etc.) and for the fixing of these same products. However, higher percentages of additives can affect the durability of materials, especially in wet environments. According to the results found in this study, it is recommendable to not exceed a 16% of addition to the gypsum matrix.

This study paves the way for other studies, such as the use of this waste in cement matrix applications or in clays to improve their properties, conduct a study using other percentages, and why not conduct a long-term durability study to find out how well the composite can resist humidity.

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