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Study of mechanical and hygrothermal properties of agglomerated cork

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1. Introduction

Cork is a natural material of cellular structure having very interesting properties, which are low density, great dimensional compressibility, good acoustic, vibration absorption, heat insulation, chemical stability and longevity [1]. The cork cells are composed mainly of suberin, lignin (40% and 22%, respectively), and of cellulose of 9% [2]. Several scientific studies related to the oak cork (growth and production of cork), natural cork (biological and chemical properties and qualities of cork), agglomerated cork (mechanical and hygrothermal characterization) are reported in the literature. With regard to natural cork, the scientific studies are related to the influence of temperature and its environment (wet or dry) on its mechanical and hygrothermal properties. The natural cork immersed in water absorbs in all the three principal directions (axial, radial and tangential). The absorption of water in the walls of cells and noncommunicating cork causes its expansion. The work [3] shows that, the water absorption capacity of cork is twice more significant in water carried at 100°C than in water at ambient temperature. When natural cork is heated at high temperatures, the loss of mass is 15% at 200°C and 62% at 350°C, beyond this temperature, the structure of the walls of cells are damaged considerably [4]. Ben Abdallah et al. [5] studied the influence of the heating of cork in a wet medium on variation of mass of Tunisian natural cork. The cork samples are heated at 100°C in the water vapour for about 3 hours then cooled and then these samples are dried in oven at 70°C for 3 hours. The results show a reduction in the density following vaporization of the water contained in the cork. For Tunisian natural cork the thermogravimetric analysis (TGA) carried out at 180°C shows a loss of mass of 4% in the first 10 minutes then 0.3% up to 30 minutes. At 220°C the loss of mass is increasing up to 30 minutes to reach 7.16%, at the end the colour of cork became brown dark. The SEM observations show that, the walls of the cells which are in the beginning corrugated became almost linear (straight) under the effect of heat, these swellings are responsible for the expansion of cork, the mass decreases following the evaporation of the water contained in cork [6].

In the mechanical field, the compression test carried out on natural cork is characterized by three phases: an elastic part corresponding to elastic bending of the walls of cells up to 5% of deformation, a stage (forced stable) corresponding to the compression of the walls of cells between them up to 60%, to 80% of the breaking stress, there is a crushing and a collapse of the cells until the rupture, this behavior is identical in the three principal directions of cork [7]. At ambient temperature, the tensile tests on natural cork show a larger tensile module in the radial direction of 60% compared to the tangential direction [8]. Strong ones et al, observed in their experiments the Poisson's ratio in the three directions (axial, radial and tangential) up to 30% of deformation compared to the breaking stress, the Poisson's ratios in the axial and radial directions are equivalent [9].

Contrary to natural cork and its limited field of application, the industrial cork covers a wide area up to thermal and acoustic insulations for space vehicles [10, 11]. Agglomerated cork is obtained from granulated cork compressed in an autoclave and crossed by a water vapour flow at 350°C [12, 13]. Several scientific studies are made, on black agglomerated cork produced in Portugal. Work of Gil presents the influence of the density on the acoustic properties of the black agglomerated cork panels. The low density cork has maximum of insulating capacity due primarily to the high quantity of air contained in the cells [14]. Lastly, several industrial studies on transformed cork are carried out but within the commercial framework. The development of sandwich panels by infusion in "one shot" presents many advantages (simple, inexpensive, allows to produce large panels); on the other hand, it cannot prevent the resin *LY 5052* from penetrating inside cork. Within the framework of this study, mechanical characterization and hygrothermal analysis of two types of white agglomerated cork of different density produced in Algeria is presented. Moreover, sandwich panels produced using the cork plates are studied in order to show the influence of the manufacturing process (infusion). The goal of the mechanical characterization is to identify using experiments the shear modulus as well as the failure stress of cork plates and to see the influence of their density as well as the presence of resin in cork material on the intrinsic characteristics. With regard to the hygrothermal study, the influence of the temperature on the variation of mass of the various types of cork is analyzed for large samples using drying oven and small samples using TGA. The capacity of absorption of the various types of cork is also studied.

2. Experimental study

Within the framework of this study two types of cork are considered and are classified based on their density (190 kg/m^3 and 270 kg/m^3). The acoustic and thermal tests showed that *C190* is the best in the acoustic field and *C270* is the best in the thermal field [15]. For each type of the cork two batches were studied. The first batch is of specimens made of the cork and the second batch is a sandwich panel made of the cork. The samples of cork plates are referred as *C190* and *C270*; on the other hand sandwich panels made of the cork are referred as *C190(R)* and *C270(R).* In the case of the sandwich panels the skin material is made of jute/epoxy [16].

2.1. Mechanical tests

Shear tests are carried out in accordance with standard NF EN12090 on both types of corks. The modulus of rigidity is calculated in two orthogonal planes of the agglomerated cork plate. Eq. (1) is used for the calculation of *Gxy* and *Gxz*.

$$G = \frac{d \times tan\, a}{A} \quad (1)$$

where A is length L x width B (mm²); $tan\, a$ is F/e (difference in force by difference in displacement in the elastic zone), d is width (mm) of the plate and G is shear modulus (MPa).

The samples tested are having dimensions 250 x 50 x 30 mm. Fig. 1 shows the assembly used for the shear tests. The cork plates are fixed on two rigid metal supports. These supports are fixed on the jaws of the Zwick tensile testing machine. The rate of travel of the jaw is of 2mm/min. During loading stress versus displacement are recorded (Fig. 1).

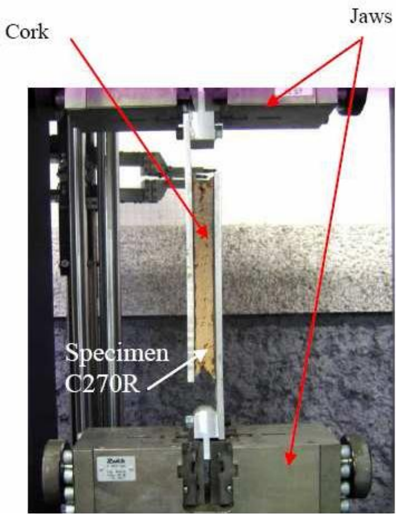


Fig. 1 Shear test specimen on tensile testing machine

2.2. Hygrothermal study

2.2.1. Drying of cork in oven

The analysis of variation of the cork mass versus time at a temperature of 100°C using oven for 24 hours duration is carried out. The samples of cork tested are having the following dimensions 250 x 250 x 30 mm. The samples are weighed before the test and thereafter, samples are weighed for each one hour during 24 hours to quantify the loss of mass.

2.2.2. Thermogravimetric analysis

TGA is carried out in an atmosphere controlled environment on cork samples of dimensions 3 x 3 x 5 mm. The cycle of temperature imposed on the samples is as follows: rise in the temperature of the cork from 25°C up to 180°C with a speed of 40°C per minute and once 180°C is reached, the cork is held at 180°C for about 30 minutes, then the temperature was reduced to 25°C at the speed of 40°C per minute. In order to see the influence of the maximum temperature on the behavior of the cork samples, a similar cycle is carried out up to 220°C.

2.2.3. Test of water absorption capacity

These tests are carried out in accordance with standard NF EN 1609 in a clean room at controlled temperature and moisture. Table represents the initial mass m_0 of specimens tested before the immersion. Thereafter the specimens partially immersed in water for 24 hours with the help of weight so that the specimen will not float on water. After 24 hours of immersion in water, the samples were drained for 10 minutes (The samples were placed vertically as shown in Fig. 2). Eq. (2) gives the capacity of absorption of water *Wp* (kg/m^2).

Table

Values of the corks masses before immersion in water

Types of cork	Initial mass $\mathbf{m_0}$ (kg)
C270	371.4 x10^{-3}
C190	314.5 x10^{-3}
C270(R)	100.3 x10^{-3}
C190(R)	113.7 x10^{-3}

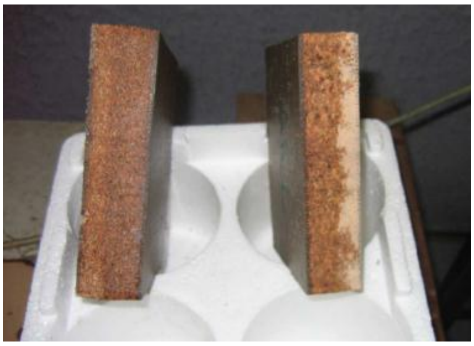


Fig. 2 Draining of samples for 10 minutes

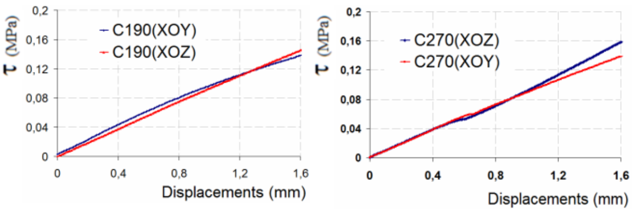
$$W_p = \frac{m_{24} - m_0}{A_p} \quad (2)$$

where W_p is absorption capacity (kg/m²); m_0 is initial mass (kg); m_{24} is mass after 24h (kg); A_p is (surface) length x width (m²).

3. Results and analysis

3.1. Shear tests

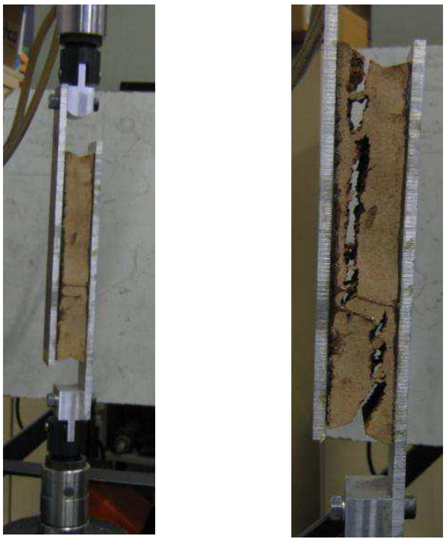
The Figs. 3, a and b shows the behaviour in shear in planes *X0Y* and *X0Z* for the cork *C190* and *C270* respectively. For *C190* cork, the linear elastic phase in planes *X0Y* and *X0Z* is observed for a stress of 0.12 MPa. The modulus of rigidity calculated in these two planes is about 2.6 MPa. Beyond 0.12 MPa one notes a nonlinear behaviour until the rupture. The maximum stress of rupture is $\tau = 0.3$ MPa in plane *XOY*, which is lesser (24%) when compared to plan *X0Z*. With regard to cork *C270*, one can note an identical linear behaviour for a stress of 0.12 MPa. The modulus of rigidity calculated in this zone $GX0Y \approx GX0 = 2.62$ MPa. Beyond this stress nonlinear behaviour is observed until the total rupture. The maximum stress of rupture is 0.33 MPa in plan *X0Y*, which is 20% larger when compared to plane *X0Z*.



a b

Fig. 3 Stress versus displacement for the shear test in the planes *X0Y* and *X0Z*, (a) *C190*, (b) *C27*

Fig. 4 shows the first (elastic zone) and final stage (rupture of cork) of the cork tested. The rupture of the cork is identical to the work of Reis et al. [17]. The modulus of rigidity of agglomerated cork of 11 mm thickness and 270 kg/m^3 of density was found by Reis et al. is 4.86 MPa for pellets of 1 mm to 4 mm.

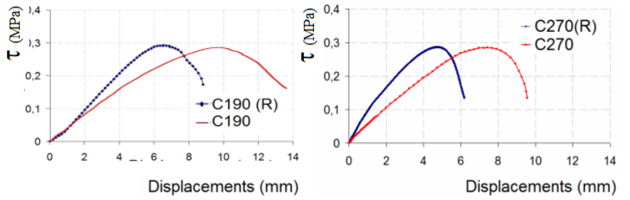


a b

Fig. 4 Typical mechanical behaviour of agglomerated cork during shear test (a): elastic domain, (b): final failure

Fig. 5 represents the curves of the stress versus displacement during the shear tests in plan (*X0Y*) of the cork *C190, C190(R), C270* and *C270(R).* In the Fig. 5, a, the maximum breaking stress of 2.9 MPa is identical for both *C190* and *C190(R)* corks. The modulus of rigidity is of 1.54 MPa for *C190* and 2.75 MPa for *C190(R)*, which is an increase of 78% when compared to *C190, C190* has a linear behavior up to 6.5 mm of displacement then a nonlinear behavior until rupture. Cork *C190(R)* has a linear behavior up to 5 mm of displacement then a nonlinear behavior until the rupture, this behavior can be related to the presence of resin inside the cork because of its larger granularity (3 to 5 mm), indeed, the presence of large pellets supports spaces between grains what allows the resin inside the cork and makes it harder but fragile.

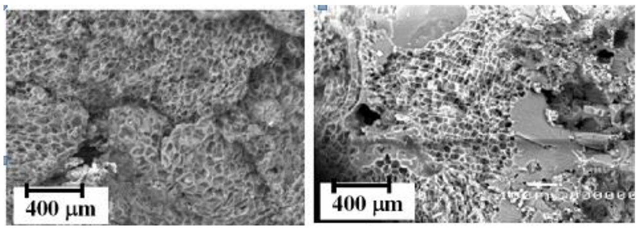
We notice in the Fig. 5, b that the modulus of rigidity calculated in the linear elastic zone is 2.25 MPa for *C270* and 3.08 MPa for *C290(R)*, which shows an increase of 37% when compared to *C270*. This increase can be again related to the presence of resin in *C270*. The modulus of rigidity of the epoxy resin is about 3 GPa. It is also noted, that the maximum stress of rupture of about 2.9 MPa which is identical for *C270* and *C270(R)* cork material.



a b

Fig. 5 Stress versus displacement during shear test in (*XOY*) plan of specimens, (a) *C190* and *190(R)*, (b) *C270* and *C270 (R)*

The observations using *SEM* show the presence of resin in the cork. The quantity of resin in cork depends on its nature. Figs. 6, a and b shows the space in the cork and resin penetration, which explains the rigid behavior of the cork with resin in shearing test compared to the agglomerated cork without resin.



a b

Fig. 6 *SEM* Observations of cork *C270* and *C270(R)*, (a) random distribution of granules, (b) presence of resin in cork *C270(R)*

3.2. Hygrothermal analysis

3.2.1. Analysis of variation of mass of cork after drying out in oven

The Fig. 7, a shows the variation of the mass of *C190* cork with time of drying at a temperature of 100°C in oven. *C190* cork loses in the first 10 hours around 5% of its mass then it is stabilized, *C190(R)* loses 3.3% in the first 10 hours then 0.7% up to 24 hours. This difference in loss of mass can be explained by the fact, that the presence of resin in cork *C190(R)* plays a role in moisture absorption.

It is noted that *C270* cork loses more mass than cork *C270(R).* In the first 4 hours *C270* loses 4% of its initial mass, where as after 10 hours the variation of its total mass compared to its initial mass is only 4.5%. Beyond 10 hours of drying in oven, no variation of mass is observed. With regard to material *C270(R)*, it loses a mass greater than *C270* material in the first hour. In this situation, one can note 2.3% of mass lost for *C270(R)* and 1.2% of mass lost for *C270*. At the end of 6 hours, cork *C270(R)* loses 3.5% of its initial mass, thereafter no variation of mass was observed. The difference in loss of the mass is slightly less than that of *C190* and *C190(R)*, this can be related to the structure of these two types of cork. It is found that if the cork material is dense, it is less sensible to moisture.

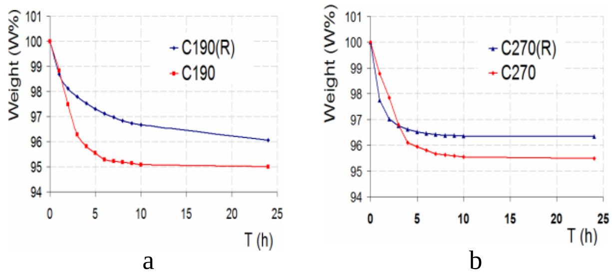


Fig. 7 Variation of the mass of cork *C190*, *C190(R)*, *C270* and *C270 (R)* according to the warming time at 100°C

3.2.2. Analysis of variation of mass of cork during TGA test

The Fig. 8, a shows the variation of mass expressed in percentage for agglomerated cork (*C270*) carried at 180°C during 35 minutes in TGA. A significant amount of loss of mass in the first 5 minutes (3.84%) is observed. Between 5 to 35 minutes the mass varies slightly (0.84%). The total loss of mass at the end of 35 minutes is 4.68%. Similar work of Ben Abdallah et al. on Tunisian untreated cork shows a loss of mass of 5% in the first 8 minutes and then a stable behaviour up to 30 minutes [5]. During the first 5 minutes at 220°C, the variation of the mass of *C270* cork is (4.13%). After the first 5 minutes the loss of mass of the cork continues and at the end of 35 minutes, the cork lost a total mass of 7.16% (Fig. 8, a).

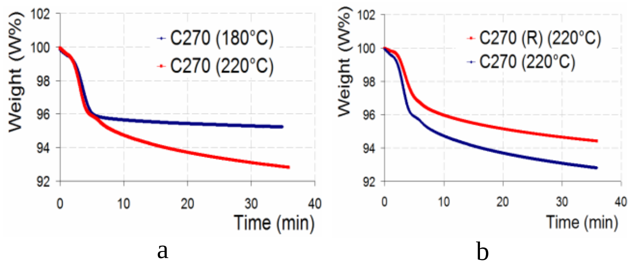


Fig. 8 *TGA* of cork, (a): *C270* at 180°C and at 220°C, (b): *C270* and *C270(R)* at 220°C

The Fig. 8, b represents the variation of the mass expressed in percentage of the *C270* and *C270 (R)* carried at 220°C. Loss of mass is different for the two types of the cork tested. Although the loss of mass is high during the first 5 minutes, the amount of loss is not the same for *C270 (R)* and *C270*; it is 5.39% for *C270 (R)* and 6.78% for C270. From 5 minutes up to 35 minutes the loss of mass is continuous and steady. This variation of mass can be explained by the fact that *C270* cork presents a higher percentage of presence of air between the grains, in cork *C270 (R)* the presence of resin inside cork prevents the mass from decreasing considerably. At 220°C the cork changes to maroon dark colour, which is similar to the results of Ben Abdallah et al. [5].

3.2.3. Analysis of absorption of water

Histogram in Fig. 9 shows the variation of the capacity of water absorption *Wp* (kg/m²) of the *C270*, *C190* corks at partial immersion condition in a vessel during 24 hours. We notice that the capacity of absorption of *C270* cork is much higher when compared to *C190* cork. *C270* has a capacity of absorption of about 4.86 kg/m², where as *C190* is only about 0.38 kg/m². This difference is certainly related to there granulometry of the two types of corks tested. Fig. 10 shows the air voids of *C190*, which is larger than that of *C270*. More the proportion of air voids, water absorption capacity of the cork is less. It is also noted that the presence of resin in material *C270 (R)* led to a capacity of absorption 26 % larger than that of *C270*. This increase can be explained by the fact that low porosities can be filled by a small quantity of resin (Fig. 11, a) which permits a good retention of water. However, a great quantity of resin fills the bigger sizes of porosity of *C190* (Fig. 11, b) which results in a weaker capacity of absorption for materials *C190 (R)*. After 24 hours of immersion of water, *C270* had a significant deformation; however *C190* cork did not change its form. Fig. 12 shows the agglomerated cork *C190*, *C270* tested for water absorption.

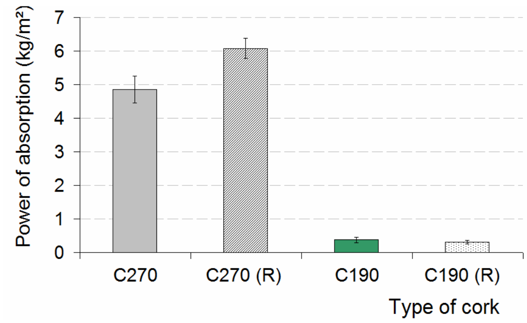


Fig. 9 Histogram of the capacity of absorption of the cork *C270, C270 (R), C190 and C190 (R)*

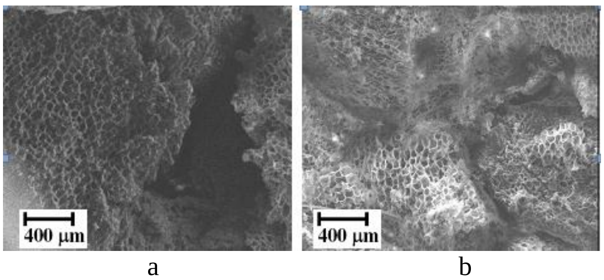


Fig. 10 *SEM* photographs showing the sizes of porosity of corks: (a) *C190*, (b) *C270*

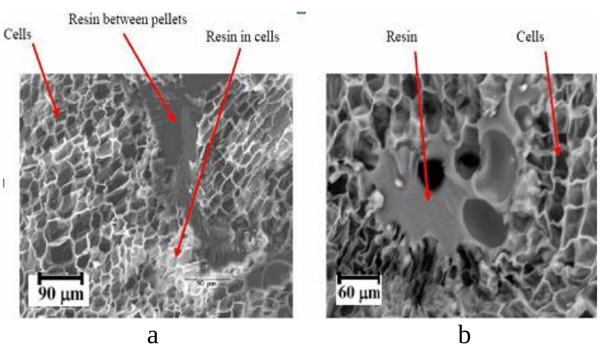


Fig. 11*SEM* photographs of agglomerated cork (a): *C190 (R)* and (b): *C270(R)*

a b

Fig. 12 Corks tested for water absorption, (a) *C190* and (b) *C270*

4. Conclusion

In this paper two of types of agglomerated corks (medium and low density) and sandwich panels are studied for its shear strength and hygrothermal properties. This study took into account the influence of presence of resin inside the cork of sandwich panel of cork. During shear tests, presence of resin in the *C220(R)* cork showed an increased modulus of rigidity of 1.8 times compared to the *C220* cork and of 2.8 times that of *C190*. In hygrothermal analysis, the influence of the temperature on the variation of the mass of cork with and without resin was observed. At 100°C, 180°C and 220°C, cork loses a significant mass in the first phase; this loss is not identical for the various types of the cork with or without presence of resin. The water absorption capacity showed that the various types of the cork do not behave same manner in wet medium, further it is noted that the variation of the density of cork and the presence of resin influence the water absorption capacity. These results highlight the influence of manufacturing process on the cork panels made of infusion process, which shall help the designers to simulate cork panels during design of structures.

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AGLOMERATINIO KAMŠČIO MECHANINIŲ IR HIDROTERMINIŲ SAVYBIŲ STUDIJA

R e z i u m ė

Kuriant daugiasluoksnes detales, pagamintas iš kamščiamedžio žievės granulių infuzijos metodu, negalima apsisaugoti nuo dervos įsiskverbimo į medžiagą. Straipsnyje apibūdinama dervos įtaka gryno kamščio, aglomeruoto prieš daugiasluoksnę gamybą ir po jos, mechaninėms ir hidroterminėms charakteristikoms. Tyrinėti dviejų tipų kamščiai, apibūdinti jų tankiu: C270 – 270 kg/m³ ir C190 –190 kg/m³. Pažymėtina, kad C270 tamprumo modulis yra 20% didesnis už C190 modulį. C270 tipo šlyties modulį derva padidina 47%, o C190 tipo modulį – 64%. Hidroterminė analizė parodė, kad džiovinant krosnyje 100ºC temperatūroje, kamščių masė per pirmas septynias valandas gerokai sumažėja. Per pirmas penkias termogravimetrinio tyrimo 220ºC temperatūroje minutes nustatytas ryškus masės sumažėjimas.

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STUDY OF MECHANICAL AND HYGROTHERMAL PROPERTIES OF AGGLOMERATED CORK

Summary

The mode of development of sandwich panels with cork core by the method infusion cannot prevent the resin from penetrating inside cork. This study characterizes white cork agglomerated before and after the development of sandwich panels to see the influence of the resin on the mechanical and hygrothermic characteristics of cork. Two types of cork are considered, they are characterized by their density of 270 kg/m^3 (*C270*) and of 190 kg/m^3 (*C190*). One notes that the modulus of rigidity of *C270* cork is 20% larger than that of *C190*.The presence of resin increases also the module of shearing 47% for *C270*, 64% for *C190*. The hygrothermic analysis out of drying oven at 100°C shows a fall of significant mass in the first 7 hours. The analysis thermogravimetry at 220°C, the fall of significant mass is recorded in the first 5 minutes.

Keywords: mechanical and hygrothermal properties, agglomerated cork.

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