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Investigation of the Effect of Cutting Directions on the Improvement of Mechanical Parameters of Treated Cork by THT: Experimental Measurement, Modelling and Optimization of Mass Transfer

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1. Introduction

Cork is a hygroscopic biomaterial characterized by a very noticeable anisotropic structure in both scales macroscopic and microscopic. Its content over time depends on climatic conditions, the environment and the nature of the soil as well [1, 2]. In addition, it is very sensitive to the seasonal distribution of rainfall and the amount of precipitation [3]. However, its anisotropic physical properties have an effect on the mass diffusion, which depends on its porosity and tortuosity because its ecological qualities of cork continue making it a premium product [4]. Therefore, it is necessary to examine the material in the three orthotropic directions in transient mode to predict not only its insulation quality and its durability but also to optimize the amount of energy required for the treatment cycle to improve the diffusion property to obtain a new material that meet the industrials need for insulation purpose.

Many researches have been conducted to study the mass transfer in various biomaterials in their raw state [5], knowing that some of them are very costly and take time to get results [6, 7]. They are distinguished by studying the effect of one or two parameters simultaneously.

In this paper, we focused to study the kinetics of the solute desorption in cork taking into account the cutting directions of native cork and cork treated with THT and boiling. Experiments are conducted to provide a significant improvement in texture for optimal D_{app} considering the porosity and tortuosity. Similarly, the type of the solute will be diversify in connection with the diffusing chemical species unlike what have been carried out with pure water or alcohol for natural biomaterial in previous studies [8, 9]. Typically, the D_{app} is determined using the indirect method of conductimetric measurement [10] on the basis of the kinetic of the solute desorption (NaCl '0.7M'), unlike to studies that has been carried out with gas [11, 12].

The solute diffuses through the anisotropic porous medium "the cork in the form of slab " to the external medium in different directions (radial, longitudinal and tangential). The model of the kinetic desorption developed in this instance is calibrated with the experimental data to determine the D_{app} according to the direction of the diffusion in the sample for both states natural and treated. The accuracy of the results is improved by using the Bat-Algorithm method.

2. Experimental protocol

The cork used in this study is grown in Skikda, Algeria, collected from the same tree to keep the homogeneity and the quality class as much as possible according to [13, 14], knowing that the Mediterranean region is a very virulent area of climate change [15, 16]. The experimental methodology consists in preparing thin plate-shaped cork samples, with an average lateral surface of 4.4×10^{-2} m^2 and $2l = 3 \times 10^{-3}$ m in thickness, on the basis of the cutting direction as shown in Fig. 1 to ensure that the flow of the diffusing solute could be measured in different directions (Fig. 2). The samples are boiled and then followed by a high temperature heat treatment (THT) under an inert gas (Argon) in programmable furnace type NABERTHERM 'More Than Heat 30-3000°C' (Fig. 3).

Fig. 1 Picture of the used cork sample

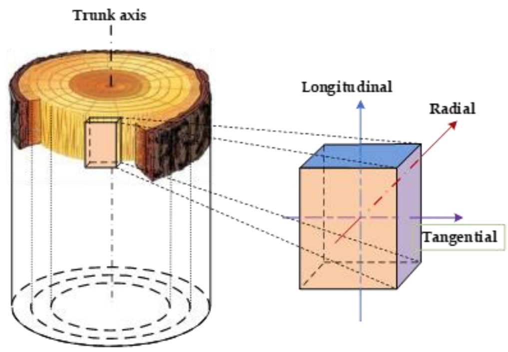


Fig. 2 Different directions of transfer

Fig. 3 High temperature treatment furnace 'Laboratory Military Academy Polytechnic'

The treatment cycle illustrated in Fig. 4, which has been inspired from the cycle proposed by [10], it is worth mentioning that this cycle brings improvements and makes the cycle more optimal. It is characterized by a level of homogenization (170°C) and a heating rate of 1°C/min at the beginning and 5°C/min after the homogenization level with the shortening of the heating process at the end of the treatment to 5 min. This heat treatment cycle gave us good results with less treatment energy.

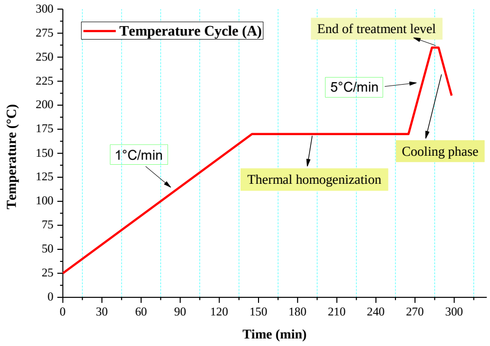


Fig. 4 Heat treatment cycle (A) of cork samples

The samples are weighed and impregnated under vacuum in NaCl (0.7M) solution. The samples are then introduced in distilled and ionized water, with a volume of 10^{-3} m^3. The instantaneous concentration is measured indirectly by a conductimetric analysis of the aqueous solution. It is worth mentioning that the different transfer directions are taken in consideration in each process.

3. Modeling

It is worth noting that the cork sample with initial solute concentration c_{i0} is immersed in stirred medium (water) characterized by an initial zero concentration. To simulate the mass transfer phenomenon some assumptions are adopted: the temperature of the medium is almost constant and the mass transfer in the boundary is much quicker. Thus, the surface concentration (c_{ip}) remains constant and can be identified to the equilibrium concentration (c^*) when the diffusion process is reached. The mathematical model is derived from a mass balance of the diffusing chemical species (i) applied to a control volume (dv) for which the statement of the mass balance may be made:

$$\begin{pmatrix} Molar\ \ flow \\ for\ \text{i in (dv)} \end{pmatrix} - \begin{pmatrix} Molar\ \text{flow of i} \\ out\ \text{from (dv)} \end{pmatrix} + \begin{pmatrix} Molar\ \text{formation rate of i} \\ from\ \text{chemical reactions} \end{pmatrix} = \begin{pmatrix} \text{Time rate of molar change} \\ of\ \text{i within (dv)} \end{pmatrix}. \tag{1}$$

In the absence of any chemical reaction and negligible contribution of convection within the control volume the mass balance takes the form:

$$\begin{pmatrix} Accumulation \\ term \end{pmatrix} - \begin{pmatrix} Diffusion \\ term \end{pmatrix} = 0. \tag{2}$$

Thus:

$$\frac{\partial c_i(\zeta,t)}{\partial t} - D_{iapp}\left[\frac{\partial^2 c_i(\zeta,t)}{\partial \zeta^2} - \frac{\beta}{\zeta}\frac{\partial c_i(\zeta,t)}{\partial \zeta}\right] = 0, \tag{3}$$

where: c_i is the diffusing concentration; D_{iapp} the apparent diffusion coefficient; ($2l$) thickness and ($\beta = 0$).

The mass diffusion equation of the model is given as follows:

$$\left.\begin{array}{ll} D_{iapp}\left[\dfrac{\partial^2 c_i(z,t)}{\partial x^2} - \dfrac{\partial c_i(z,t)}{\partial t}\right] = 0 & \\ c_i(x,t)\Big|_{\substack{x>0 \\ t=0}} & = c_{i0} \\ c_i(x,t)\Big|_{\substack{x=l \\ t>0}} & = c_{ip} \\ \dfrac{c_i(x,t)}{\partial t}\Big|_{\substack{x=0 \\ t>0}} & = 0 \end{array}\right\}. \tag{4}$$

By applying those initial and boundary conditions the final solution of the model is therefore:

$$\frac{C_i(x,t) - C_{i0}}{C_{ip} - C_{i0}} = 1 - \frac{4}{\pi}\sum_{n=0}^{\infty}\left\{\frac{1}{2n+1} sin\left[\frac{(2n+1)\pi}{2l}x\right] exp\left[-\frac{(2n+1)^2\pi^2 D_{iapp}}{4l^2}t\right]\right\}. \tag{5}$$

Regarding the reduced concentration, the instantaneous quantity of the diffusing mass through the wall ($x=l$) can be estimated by: $\frac{m_t}{m_\infty} = \frac{c_t}{c_\infty}$, where: m_t mass of the substance released at time t and m_∞ mass substance transferred after total desorption from the shaving after an infinite time. From where the final model equation could be written as follow:

$$\frac{m_t}{m_\infty} = 1 - \frac{8}{\pi^2}\sum_{n=0}^{\infty}\left\{\frac{1}{(2n+1)^2}\ exp\left[-\frac{(2n+1)^2\ \pi^2\ \mathrm{D}_{iapp}}{4l^2}\ \mathrm{t}\right]\right\}. \tag{6}$$

4. Results and discussion

The obtained results show that the heat treatment has affected the shifted and random arrangement of cellular basis because the ridged surface of the sidewalls has an impact on the value of the diffusion coefficient due to the change in the tortuosity and the porosity of the diffusion channels.

Indeed, the crumpling of the cell walls of the samples treated with THT was observed by SEM (Fig. 5). This histological morphology is physically translated by the modification of porosity and tortuosity.

In contrast to the classical studies carried out on native boiled or / and bleached cork [17, 18], our investigation is reinforced by an infrared (IR) spectral analysis of samples for both native and treated cork.

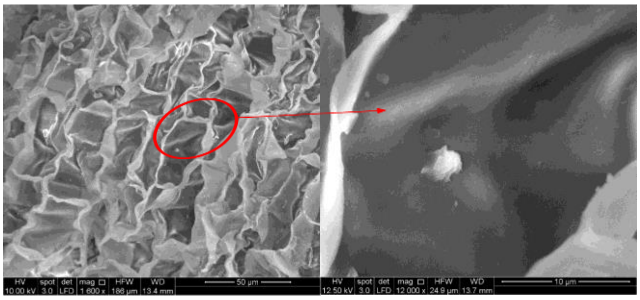


Fig. 5 Cell structure of cork after THT observed by SEM

Fig. 6 shows the IR spectra for both treated and native samples in the frequency range between 3.2x10^{-3} and 3.4x10^{-3} m^{-1}. This band is associated with the vibration of elongation of hydroxyl groups (OH) by the intermolecular interactions of hydrogen bridges. In fact, in the treated sample, the absorbance is much lower (about half) than the native sample.

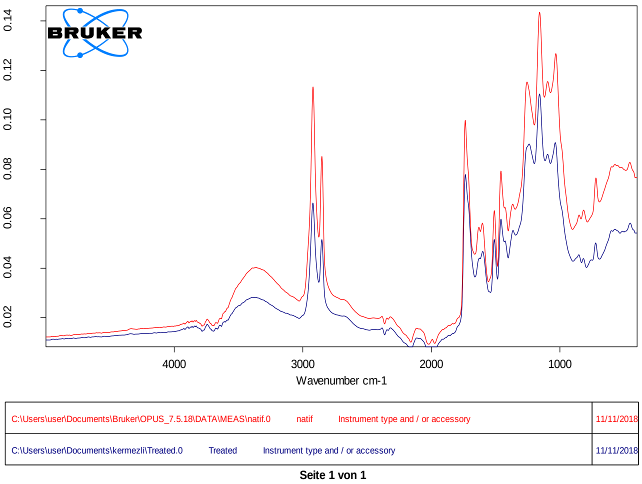


Fig. 6 IR-FT spectrum of native and treated cork samples

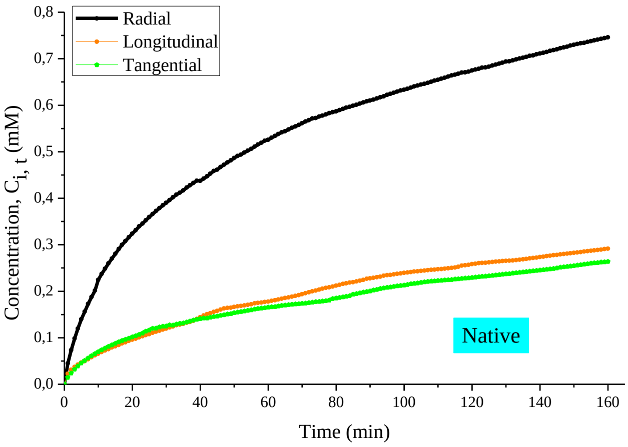
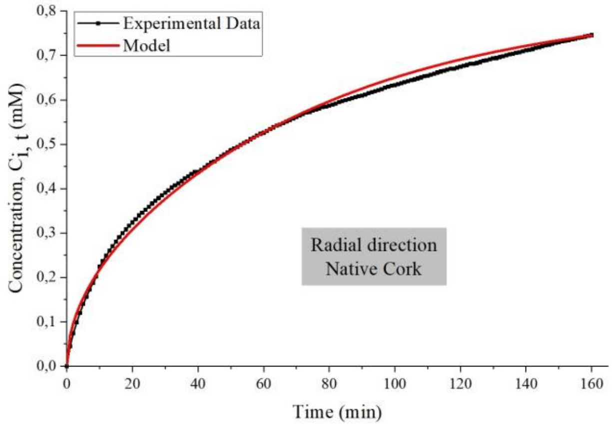


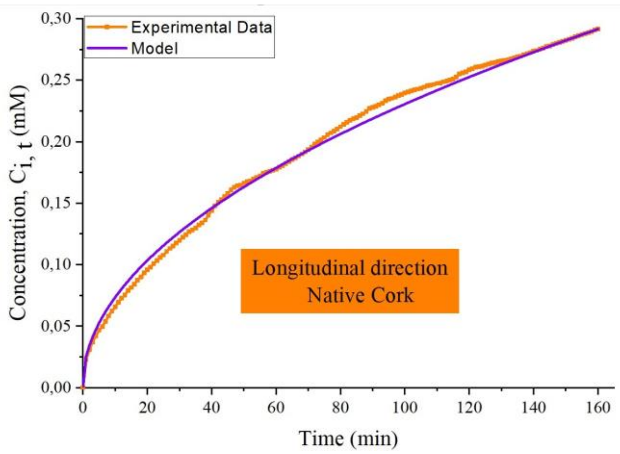
Fig. 7 Variation of the concentration in the native cork according to the different directions of transfer

From Fig. 7, the instantaneous variation of the NaCl chemical tracer is much faster in the radial direction than in the longitudinal and tangential directions. indeed, according to the morphology of cork the radial direction is the preferred path for the diffusion of gases, water and nutrients because of not only the presence of lenticular channels (pores) parallel to the radial direction of the plant growth but also the well-arranged placement of the cells in that direction.

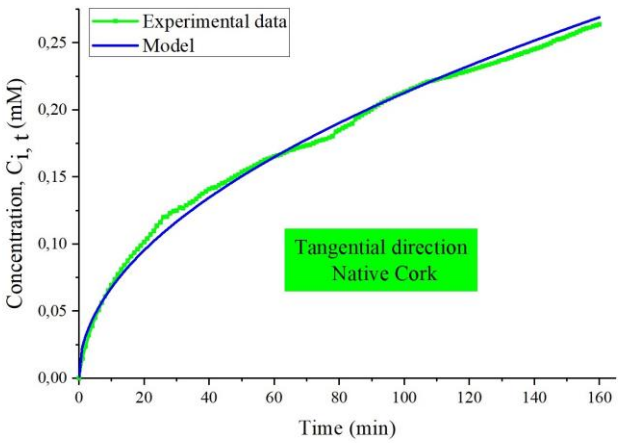
The mass transfer velocity in the longitudinal direction is slightly greater than in the tangential direction for the native cork, due to the structural anisotropy of the material, which influences the kinetics desorption. This result is in good agreement with the results presented by Mouchot [19].



a



b



c

Fig. 8 Calibration of the experimental results with the model for the different transfer directions

Fig. 8, a – c shows the consistence of the model with the experimental data of the instantaneous concentration measured indirectly in the external medium in three directions. The D_{app} is estimated through the calibration of the experimental results with those obtained from the model whereas the optimization of its value is determined by using the Bat-Algorithm with a relative uncertainty value about 10^{-7} (Fig. 9).

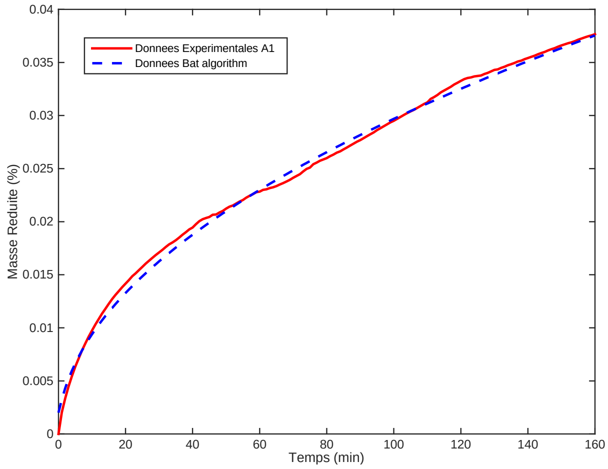


Fig. 9 Optimization of the Mass Transfer coefficient in native cork in the radial direction using BA

In addition, Fig. 10 shows the instantaneous concentration of the diffusing chemical species after heat treatment from various directions according to the treatment protocol adopted. It is worth noting that there is a similarity in the instantaneous concentration profiles in three directions. However, it is found that the diffusion coefficient of cork treated in the radial direction (D_{AR}) of cell growth, is higher than that of the other directions of cork treated. Nevertheless, in the tangential direction of cork treated (D_{AT}) is slightly larger than the D_{AL} corresponding to the longitudinal direction.

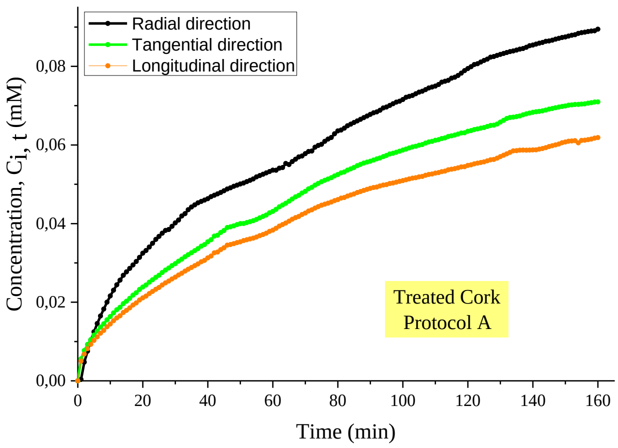
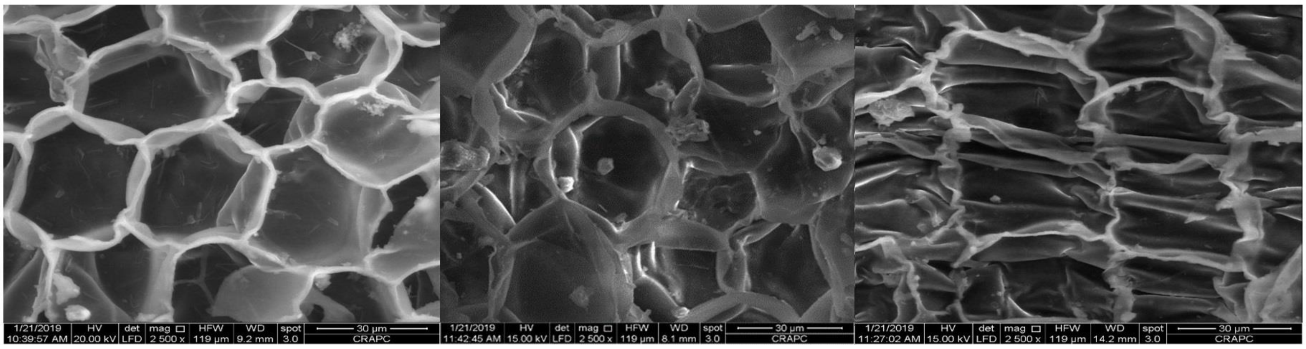


Fig. 10 Variation of the external concentration of treated cork according the directions of transfer

This result could be explained by the large crumpling of the horizontal cellular walls resulting from the THT treatment because they are particularly too thin compared to the vertical cellular walls. This effect may bring a distortion of the material tissue resulting in the increase of the tortuosity in the longitudinal direction (Fig. 11). In addition, the plots in Fig. 12 show the consistency between the experimental results of the instantaneous concentration in the external medium and those obtained with the model.

Fig. 13 illustrates the impact of cork on the longitudinal mass transfer direction. The obtained results indicate clearly that the D_{app} value in the longitudinal direction is positively affected after treatment. Indeed, the gain in term of mass insulation in the longitudinal direction after treatment by using the cycle A is 15 times compared to the native cork.

It is worth mentioning that the obtained results enabling not only to determine value of D_{app} but also the tortuosity τ on the basis of porosity ε via Eq. (7) [20].



a b c

Fig. 11 Cell wall of the cork: a) native radial direction; b) treated radial direction; c) treated longitudinal direction

$$\tau = \frac{D_A}{D_{app}}\varepsilon. \quad (7)$$

It should be noted that the porosity of cork is not equivalent to voids, as is the case with other materials, given that the absence of a correlation between density and porosity due to the pockets of pores and not connected pores [21].

In this regard, the results are summarized in Table 1 which showed that the treatment has a very positive effect on the mass insulation resulting in an improvement of the average apparent diffusion coefficient for 9 times, these results have been confirmed by [21]. The cycle used in this investigation which is optimized in time and energy causes a clear non-linear increase in the tortuosity of the treated cork compared to the native one according to the different directions. This increase is justified by both the change in the chemical groups as shown in IR spectra (Fig. 6) and the shrinkage of pores caused by THT preceded by boiling.

Table 1

Apparent diffusion coefficients and tortuosity basis on the direction of transfer

	Direction	D_{app} (m^2.s^{-1})		Tortuosity, τ	
		Native	Treated	Native	Treated
Cork samples NaCl (0.7M)	Radial	D_{NR} = 6.23x10^{-12}	D_{AR} = 5.76x10^{-13}	3.16	5.65
	Longitudinal	D_{NL} = 4.01x10^{-12}	D_{AL} = 2.70x10^{-13}	3.53	6.83
	Tangential	D_{NT} = 2.25x10^{-12}	D_{AT} = 3.67x10^{-13}	4.07	6.32

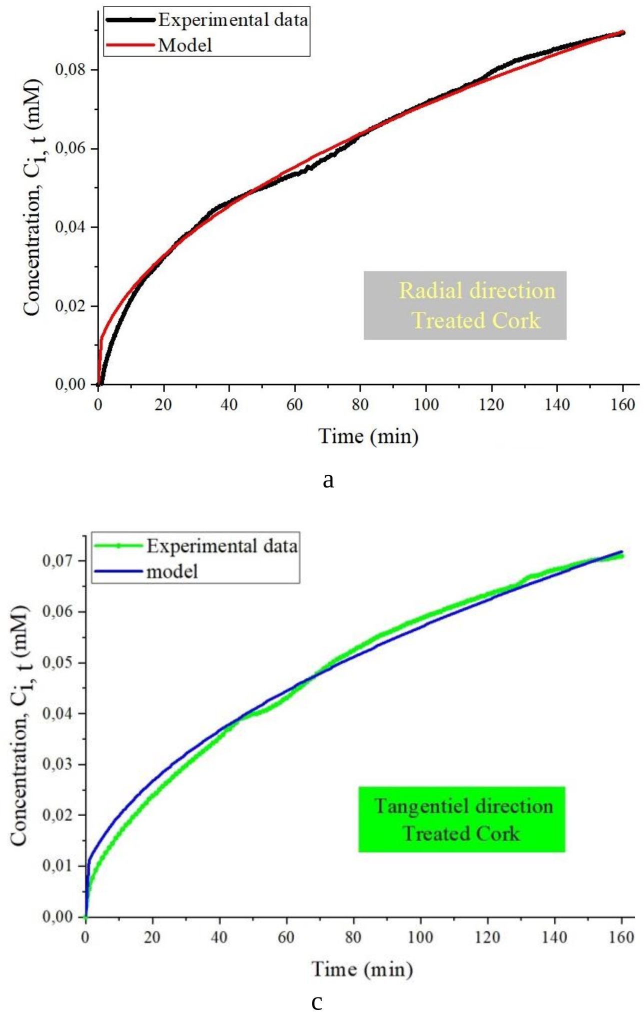


Fig. 12 Modelling of the diffusion directions effect on the concentration variation in the external medium

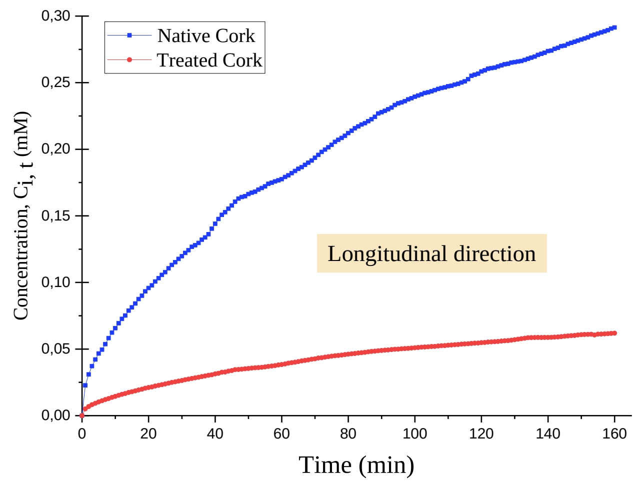


Fig. 13 Mass transfer in treated and native cork in the longitudinal direction

Table 2

Optimization Results of D_{app} according to the direction of transfer

Direction	D_{app} (m^2.s^{-1}), Native			D_{app} (m^2.s^{-1}), Treated		
	Calibration	BA	Calculation precision BA	Calibration	BA	Calculation precision BA
Radial	D_{NR}=6.23x10^{-12}	6.25x10^{-12}	2.452x10^{-6}	D_{AR}=5.76x10^{-13}	5.67x10^{-13}	1.022 x10^{-6}
Longitudinal	D_{NL}=4.01x10^{-12}	4.07x10^{-12}	3.044x10^{-7}	D_{AL}=2.70x10^{-13}	2.53x10^{-13}	1.234x10^{-8}
Tangential	D_{NT}=2.25x10^{-12}	2.19x10^{-12}	8.900x10^{-8}	D_{AT}=3.67x10^{-13}	3.79x10^{-13}	3.420x10^{-7}

Table 2 delineates the results of the D_{app} optimized by the BA algorithm proposed by [22] with a relative uncertainty value of 10^{-7}. In addition, the order of the diffusion coefficients values was maintained after being refined by optimization using Bat algorithm.

4. Conclusion

For the best utilization of cork for mass insulation purpose, the impact of the diffusion direction is investigated for both the native and treated cork by a boiling followed by a THT using an energy-optimized cycle.

The analysis of the instantaneous concentration of the solute in the medium where the sample is immersed enables to determine the D_{app} and the tortuosity. The apparent mass coefficient diffusion of the native cork for three directions is in the following order: $D_{NR}>D_{NL}>D_{NT}$=2.19x x10^{-12} m^2.s^{-1}. The heat treatment has brought a significant improvement in the mechanical structure of the biomaterial. In addition, the association of the cutting direction and the structural modifications obtained by treatment contributes to increase the mass insulation in the three transfer directions. The increasing order of the diffusion coefficients is maintained in the radial direction and inverted for the other two directions after treatment: $D_{AR}>D_{AT}>D_{AL}$=2.53x x10^{-13} $m^2.s^{-1}$, noting that these transfer coefficients have retained their order of magnitude after optimization this could be due to the shrinkage of the porous medium caused by the THT. This treatment protocol and the choice of cut direction will help manufacturers to increase the insulation of panels and stoppers made with cork leading to a new competitive material.

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INVESTIGATION OF THE EFFECT OF CUTTING DIRECTIONS ON THE IMPROVEMENT OF MECHANICAL PARAMETERS OF TREATED CORK BY THT: EXPERIMENTAL MEASUREMENT, MODELING AND OPTIMIZATION OF MASS TRANSFER

S u m m a r y

This work aims to investigate the impact of the cutting direction on the transfer properties of both the native cork and the treated by an optimized cycle at high temperature and boiling for the purpose to improve its mechanical characteristics in all three orthotropic directions. The study of the insulation performance is based on tracking the apparent mass diffusion coefficient (D_{app}) variation in order to evolve a new material with the thermal, mass, acoustic and vibratory properties needed for various applications. TGA, IR and SEM analyzes are used to confirm the enhancement of the treated cork. The D_{app} of the diffusing chemical species, NaCl (0.7M), is determined by conductimetric method. In transient mode, a mathematical model of mass transfer, which includes the diffusion parameters of the chemical species, is developed. Through the calibration of the model with the experimental measurements the D_{app} values are determined and then refined numerically by optimization using the Bat-Algorithm. The obtained results show that the treatment improves the diffusion property of the native (D_N) compared to the treaty (D_A) with the values in the order of 10^{-12} and 10^{-13} $m^2.s^{-1}$ respectively with a relative uncertainty of 10^{-7}. In addition, this investigation reveals that the radial diffusion coefficient (D_R) is significantly higher than the tangential diffusion (D_T) and longitudinal (D_L) diffusion coefficients which both are almost the same.

Keywords: cork, direction of diffusion, THT cycle, modeling, optimization.

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