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Influence of water and ethanol vapours on the compressive behaviour of natural cork during ageing

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ABSTRACT

The effect of the storage condition and time on the compressive behaviour of natural cork was investigated by performing quasi-static and Progressive Repeated Loading (PRL) tests. Cork cubes were exposed to water, water-ethanol and ethanol vapours for 3- and 12-months' storage. A significant difference for the elastic modulus was observed between samples placed in contact with water (9.6 ± 3.2 MPa) and ethanol (14.3 ± 4.2 MPa) vapours after 3 months. For cork samples in contact with ethanol a significant reduction of the elastic modulus value to 6.3 ± 0.8 MPa was noted at 12 months of storage, whereas no difference was noticeable for samples in contact with water vapour. From the PRL test performed after 3 and 12 months of storage, cork exhibited a different stored energy capability according to the storage condition. A reduction of ~40 % occurred when placed in contact with ethanol vapour, whereas it increased of ~15 % when stored in contact with water vapour. This led to the hypothesis that between 3 and 12 months of storage, ethanol accelerated the ageing of the material, leading to modification in the mechanical behaviour of cork, compared with water vapour.

1. Introduction

The outer bark of the oak tree (*Quercus suber* L.) is named cork. Its main functions are to protect the tree from dehydration and fire and to ensure gas exchange with the environment (Silva et al., 2005). The world cork production is about 200 thousand tons per year (APCOR, 2020). From the Antiquity to the present, thanks to its properties, this lightweight natural material has been used in many different applications. Indeed, cork has good chemical stability (Flores et al., 1992), low permeability to liquids (Maga and Puech, 2005), rather high barrier to gases (Crouvisier-Urion et al., 2018a) and thermal-acoustic insulation properties (Gibson and Ashby, 1997). Moreover, it exhibits outstanding mechanical properties, particularly in compression since is able to undergo large deformations, up to 80 % of nominal strain (Lagorce-Tachon et al., 2015). Cork also displays a fast and important dimensional recovery when the stress is released (Rosa and Fortes, 1988a). High quality cork is mostly intended for the wine industry and is harvested every 9 years between ~40 years and ~200 years of the oak tree's life. The manufacture of cork-based stoppers (natural and agglomerated) represents 73.5 % of the world cork production (APCOR, 2020), which corresponds to 13 billion cork stoppers per year (Amorim, 2024). Bar top cork closures are also used to seal alcohol beverages such as liquors, spirits and fortified wines. The cork extracted before ~40 years of the oak tree's life, that is unsuitable for oenology, and the cork waste from the cork stopper industry, are valorised in the construction sector accounting for 24.1 % of the world cork production, as expanded cork agglomerates for thermal insulation (Lakreb et al., 2023), concrete structure (Tedjditi et al., 2020), and anti-vibration layers (Dias et al., 2018) as well as in material engineering applications (for 2.4 % of the global production) as ablative insulator in spacecraft (Reculusa et al., 2006), helmets (Ferreira Serra et al., 2022a; Coelho et al., 2013) and medical device (therapeutic shoes) (Pereira, 2007). The mechanical behaviour of cork has been extensively studied in the literature because of its versatility in absorbing different mechanical stresses, including shock, vibration, tension, torsion and creep (Dias et al., 2018; Ferreira Serra et al., 2022b; Kaczyński et al., 2019; Anjos et al., 2010; Sarasini et al., 2019; Rosa and Fortes, 1988a). In its use as a stopper, cork is submitted to compression, first in the bottling machine and then in the glass bottleneck. For still wines, natural or agglomerated cork stoppers are firstly compressed at 35 % of strain (in the radial and tangential

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directions considering the natural cork) corresponding to decreasing of the stopper diameter from 24 ± 0.4 mm to 15.5 ± 0.5 mm in the bottling machine jaws. Secondly, the stopper bounces-back to the inner diameter of the bottleneck at 18.5 ± 0.5 mm, which corresponds to 23 % of strain. This leads to a 40 % compression in volume of the material in the bottleneck (Charte des bouchonniers liègeurs, 2006). In the case of sparkling wines, the stopper is even more compressed, at 50 % of strain in the bottling machine (reduction in diameter from 31 mm to 15.5 mm). Then, the stopper recovers to bottleneck size at 17.5 ± 0.3 mm (43 % of strain) (AFNOR, 1995). In this case, this corresponds to 70 % compression in volume of the material in the bottleneck (Crouvisier-Urion et al., 2018b). Cork is composed of phellem. This botanical term refers to closed, dead and empty cells with a polygonal base of ~20 μm edge and 25–40 μm height. The cell wall has a uniform thickness of ~1 μm. The compressive behaviour of natural cork has been extensively studied in the literature. From quasi-static compression test, cork exhibits a mechanical behaviour characteristic of a cellular material. It consists of three successive regions described as firstly the elastic-like region, then the plateau region and finally the densification region. The stiffness of cork is usually characterized by the elastic modulus, calculated from the slope of the elastic region of the nominal stress-strain curve (Rosa and Fortes, 1988a, 1988b; Gibson et al., 1981; Fortes and Teresa Nogueira, 1989; Pereira et al., 1992; Oliveira et al., 2014). It has already been shown that several factors can affect the mechanical behaviour of cork, related to the material, such as anisotropy (Gibson et al., 1981; Pereira et al., 1992; Oliveira et al., 2014), growth rate (Pereira et al., 1992), cork origin (Oliveira et al., 2014), density and porosity (Anjos et al., 2008, 2014) or related to the environment such as temperature (Rosa and Fortes, 1988b). Although cork-based stoppers have been used for the preservation of alcoholic beverages since ancient times, only few studies investigated the effect of hydration on the compressive behaviour of cork. Lagorce-Tachon *et al.* showed that the elastic modulus is strongly affected by the presence of water when cork is stored at relative humidity higher than 50 % (Lagorce-Tachon et al., 2015). The same phenomenon was observed both for natural and agglomerated cork stoppers (Lagorce-Tachon et al., 2016). In the case of ethanol, to the best of our knowledge, only scarce information regarding the effect on cork can be found in the literature. While water is sorbed on cork following a reversible physisorption mechanism (Lequin et al., 2010), in the case of ethanol an irreversible chemisorption mechanism occurred in addition to physisorption (Lequin et al., 2013).

In the context of wine storage, research on cork stoppers has mainly focused on the characterization of gas barrier properties (Lopes et al., 2006, 2007; Fonseca et al., 2013; Karbowiak et al., 2019; Echave et al., 2021; Chanut et al., 2023; Ureña et al., 2024; Suhas et al., 2025). However, limited research has been conducted on their mechanical aging. Depending on whether the bottle is stored horizontally or vertically, the cork stopper compressed in the bottleneck is in contact with the liquid phase or the vapour phase of a water-ethanol solution in which the alcohol content is at least 8.5 % v/v (IOVW, 2024). Thus, it is crucial to study the effect of the main components of wine, water and ethanol, to better understand the mechanical behaviour of the cork stopper during the wine bottling ageing.

The first objective of this work is to characterise the compressive behaviour of cork when stored under specific environmental conditions in contact with water and/or ethanol vapours. The second objective is to study the effect of time, up to 12 months of storage, on its mechanical behaviour. Both quasi-static (QS) compression and Progressive Repeated Loading (PRL) tests applied to cork previously equilibrated under specific conditions have been designed to address these questions.

2. Materials and methods

2.1. Sample preparation

High-quality natural cork stoppers (24 mm diameter, 49 mm height) without surface treatment were supplied by the company Trescases S.A. (Le Boulou, France). Cubes of 15 × 15 × 15 mm (165 ± 20 $kg.m^{-3}$) were manufactured using a micro milling machine (Kern micro GMBH, Germany). Cube dimensions were selected according to previous studies on mechanical properties of cork (Lagorce-Tachon et al., 2015; Rosa and Fortes, 1988a; Gibson et al., 1981; Anjos et al., 2014). After a two-weeks dynamic vacuum applied to remove water, cork samples were stored at 25 °C in air-tight containers under three different storage conditions: (i) in contact with water vapour close to saturation using a potassium sulphate saline saturated salt solution (K_2SO_4, Sigma-Aldrich, CAS 7778–80–5, Germany) to fix 97.3 % relative humidity (RH); (ii) in contact with ethanol vapour (≥99.8 %, CAS 64–17–5, Germany); (iii) in contact with water-ethanol vapour (using a water-ethanol mixture at 90–10 % v/v). Equilibrium was considered to be achieved when the weight variation did not exceed 0.05 wt% after 3 months of storage. This condition was referred to as t_3. A second condition after 9 more months of storage in contact with water, water-ethanol and ethanol vapours was referred to as t_{12} (3 months for equilibrium followed by 9 months storage). After equilibrium or aging phase in a specific environment, the samples were directly analysed for their mechanical properties while being maintained in their storage environmental conditions until the time of testing.

2.2. Mechanical testing

MTS criterion 45 tensile machine, equipped with a 5 kN load cell, was used to perform all uniaxial compression tests on cork cube (QS and PRL), between two parallel steel platens, along the tangential direction of cork at a crosshead displacement rate of 1 $mm \cdot min^{-1}$. Polytetrafluoroethylene (PTFE) film (0.125 mm thick) was laid on the platens to reduce the friction coefficient with the cork sample. Quasi-static compression test at t_3 and progressive repeated loading tests at t_3 and t_{12} were carried out on cork cubes for all condition tested. Mechanical tests were performed on at least 4 replicates for each condition of time and environmental storage.

2.2.1. Quasi-static compression test (QS)

Cork cubes were compressed at 80 % of nominal strain along the tangential direction to obtain the full stress-strain curve. For all samples, the elastic modulus was calculated from the slope of the nominal stress-strain curve in the elastic region in which they exhibit quasi-linear behaviour, using a specific strain interval of 1 %. Because cork is a biological material, its elastic region can be characterised by different slopes due to the adaptation of the sample with the moving platen fixed to the crosshead. For this reason, to identify the quasi-linear behaviour, the strain interval was shifted within the elastic region to calculate the elastic modulus more accurately. In addition, to study the effect of ageing of the material, the elastic modulus at t_{12} was calculated, using the same method, from the first loading curve obtained by the PRL test (5 cycles). This PRL test was carried out in the same experimental conditions (equipment, test speed) as the QS test.

2.2.2. Progressive repeated loading test (PRL)

Cork samples were compressed at five increasing strain levels, selected from the full true stress-strain curve. These strain levels are 3 % (elastic region), 18 % and 37 % (plateau region), 50 % and 58 % (densification region). They correspond to 3 %, 20 %, 46 %, 66 % and 80 % nominal strain, respectively. A detailed description about the use of nominal or true stress and strain values is reported in our previous work (Gerometta et al., 2023). First the sample was compressed up to a set displacement and then returned to its initial position. For each strain level, a loading-unloading cycle was repeated three times (referred to as repetitions *a*, *b*, *c*, respectively) before going for the next compression step, as detailed in Fig. 1a. In total, each cork cube was thus compressed fifteen times. As an example, Fig. 1b displays the corresponding first (*a*), second (*b*) and third (*c*) repetitions of the 3rd cycle, respectively. Each repetition consists in a loading (solid line) and an unloading (dashed line) curve (Fig. 1b). For each of the five strain levels and for each of the three repetitions, four parameters were determined (Fig. 1b): (1) The tangent modulus (E_t) was calculated from the true stress-strain unloading curve, following the protocol detailed in (Gerometta et al., 2023); (2) The stored energy (*W*) corresponding to the area under the loading-unloading curve of each cycle repetition and is approximated by the trapezoidal integration method; (3) The residual strain (ε_r) was calculated as the difference between the strain values corresponding to a stress equal to 0 MPa on the loading and the unloading curve; (4) The stress loss (σ_l) was determined as the difference between the maximum stress values of two successive repetitions for each cycle.

2.3. Statistical analysis

One-way analysis of variance (ANOVA) with a Tukey's multiple comparison test (p value < 0.05) was performed on data obtained from quasi-static compression test to compare the mean value of the elastic modulus calculated for cork cubes stored under different storage conditions. The same statistical test was used to compare each of the four parameters (true tangent modulus, stored energy, stress loss and residual strain) calculated from the PRL test. In all cases, the conditions for the application of a one-way ANOVA test have first been assessed, i.e. the normality of residuals, the homogeneity of variance of residuals, and the independence of measurements. For all parameters, the Student's test (p value < 0.05) was used to compare the mean values over ageing, at t_3 and t_{12}.

3. Results and discussion

3.1. Effect of ethanol and water vapor sorption on the elastic modulus over the storage period

The nominal stress-strain curves obtained from quasi-static compression tests performed on natural cork cubes in contact with water, water-ethanol and ethanol vapours are displayed in Fig. 2a, b, c, respectively. The 95 % distribution is represented by the shaded area. The mean stress-strain curve of cork (black line) is characterised by three regions: (i) an elastic region, up to 3 % of strain, in which cork exhibits an elastic-like behaviour; (ii) a large plateau region, between 3 % and 55 % of strain, in which a progressive buckling of the cell walls occurs, and the stress increases slightly, approximately between 0.2 and 1.5 MPa; (iii) a densification region, above 55 % of strain, in which the cell walls begin to collapse, and which corresponds to a sharp increase in the stress value to about 12 MPa. Regardless of the storage condition, it

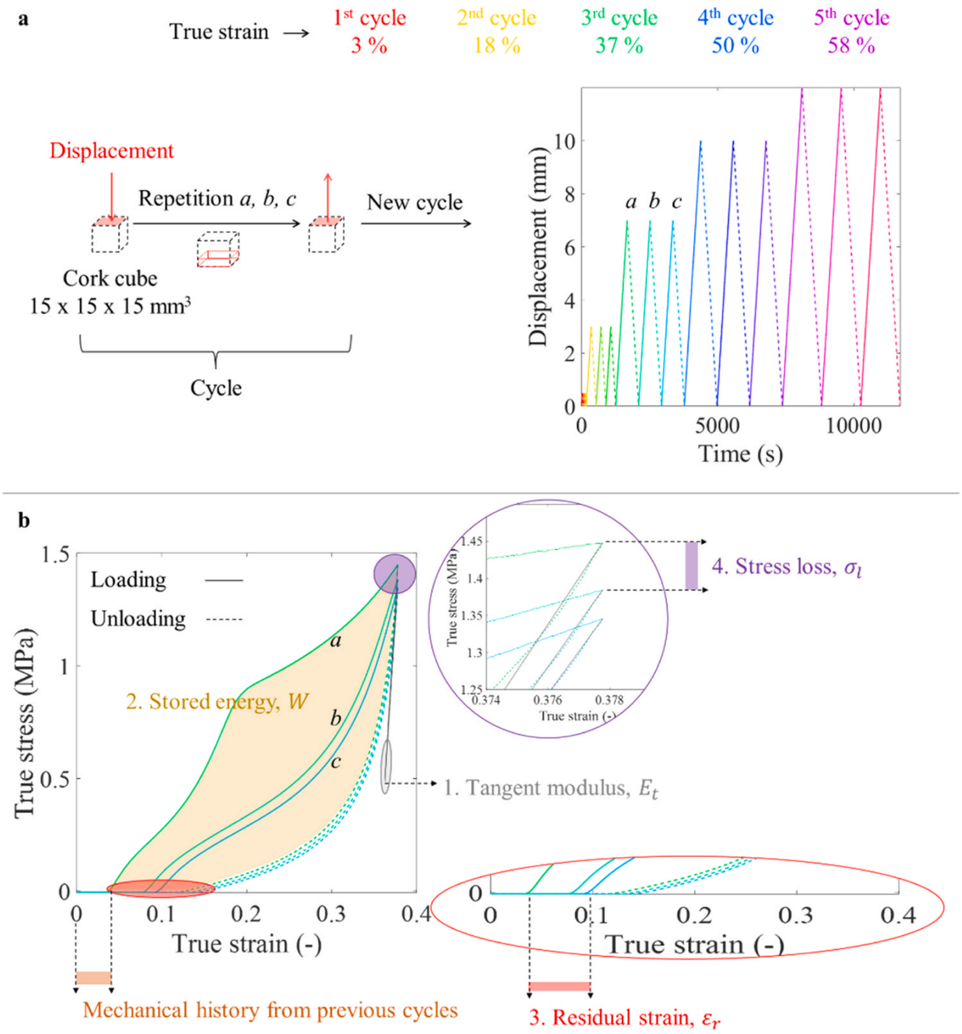

Fig. 1. (a) Schematic diagram of the PRL test: the increasing displacement values as a function of time corresponding to the five cycles applied to the cork cube are displayed on the graph. (b) PRL parameters determined for each cycle: (1) tangent modulus (E_t), (2) stored energy (W), (3) residual strain (ε_r), (4) stress loss (σ_l).

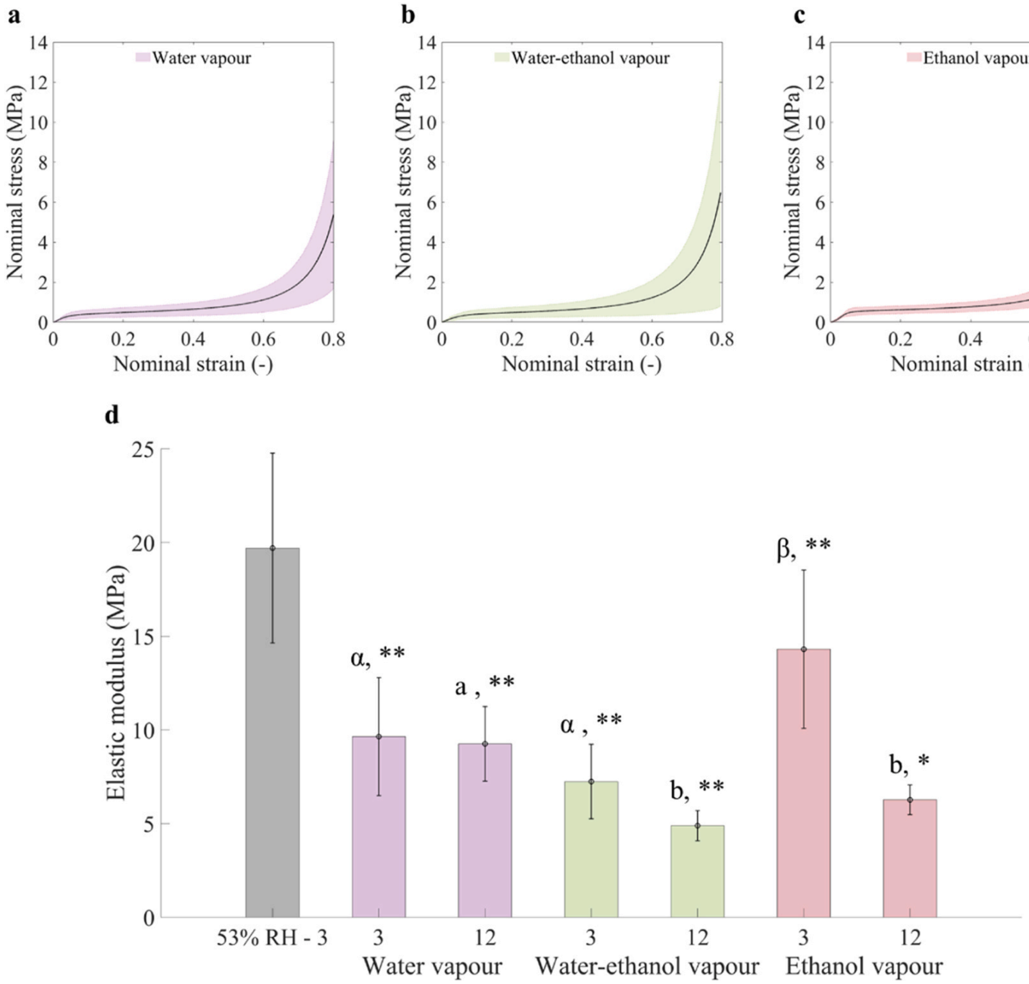

Fig. 2. Stress-strain curves obtained from quasi-static compression test performed on cork cubes previously stored up to equilibrium (for 3 months) under different conditions in contact with: (a) water vapour, (b) water-ethanol vapour, (c) ethanol vapour. (d) Elastic modulus value calculated in the elastic region of the nominal stress-strain curve from QS test (after 3 months of storage) at 25 °C and ~50 % RH ($n = 6$) (Gerometta et al., 2023). Elastic modulus values calculated from QS test (after 3 months of storage) ($n = 7$) and PRL test (after 12 months of storage) calculated from the 1st cycle ($n = 4$). Statistics: One-way ANOVA (Significant differences, with p value < 0.05, are indicated with different letters. α, β: difference between storage conditions at 3 months of storage for QS test. a, b: difference between storage conditions at 12 months of storage for PRL test). Student's t test (**, * effect of time for the same storage condition carried out with QS (3 months of storage) and the PRL test (12 months of storage)).

is noteworthy that, natural cork exhibits a rather high variability in its compressive behaviour, with nominal maximal stress values ranging from 1 to 12 MPa. This behaviour is typical of biological materials such as cork. A coefficient of variation of 30 % and 20 % was observed for samples tested at 3 months (7 repetitions) and 12 months (4 repetitions), respectively. Similar results were found in the literature, ranging from 16 % to 38 % (Gibson et al., 1981; Pereira et al., 1992; Oliveira et al., 2014; Anjos et al., 2014). For elastic modulus, an average value of 9.6 ± 3.2 MPa was obtained for t_3 cork samples placed in contact with water vapour. In a previous study, for the same storage time, an average value of 19.7 ± 5.1 MPa was found for cork stored at ~50 % RH at 25 °C (Gerometta et al., 2023). Such reduction when exposed to higher relative humidity environment is in line with previously results obtained by Lagorce *et al.* who studied the effect of hydration on the mechanical properties of cork subjected to quasi-static compression test. They observed that the elastic modulus remains constant if cork is placed in contact with water vapour, up to ~50 % RH at 25 °C. Then, a reduction of about 50 % occurred when exposed to environment with high relative humidity, from 50 % to 100 % RH (Lagorce-Tachon et al., 2015). Such mechanical behaviour is related to water sorption properties of the material. The adsorption mechanism of water molecules firstly occurs on hydrophilic sites, inducing a water-mediated hydrogen bond network between polymer chains. Secondly, the water clusters formation plasticises the cork polymers chains. This leads to a decrease in the stiffness of the material, as macroscopically evidenced by the reduction in the elastic modulus (Lagorce-Tachon et al., 2015).

No significant difference on the average values of the elastic modulus was observed for t_3 cork samples stored in contact with water (9.6 ± 3.2 MPa) and water-ethanol (7.2 ± 2.0 MPa) vapours. In contrast, samples exposed to ethanol vapour (14.3 ± 4.2 MPa) displayed significantly different values from those calculated for the other two storage conditions (Fig. 2d, statistics: α, β and appendix E.6). The presence of ethanol vapour led to an increase in the elastic modulus value, thus the stiffness of cork. After 12 months of storage, the elastic modulus obtained for samples placed in contact with water-ethanol (4.9 ± 0.8 MPa) or ethanol (6.3 ± 0.8 MPa) vapours were significantly different from that of water vapour (9.3 ± 2.0 MPa) (Fig. 2d, statistics: a, b). These results highlighted that this parameter is not affected by water vapor during the storage period monitored in this study, indeed, no significant difference between t_3 and t_{12} values was noticed, as well as for water-ethanol vapour. On the contrary, in the case of ethanol, t_3 and t_{12} values were significantly different (Fig. 2d, statistics: **, *). A possible explanation for these results lies in the different sorption phenomena on cork for water and ethanol, as described by Lequin et al. (2010). Water molecules are sorbed following a physisorption mechanism. This implies that the molecules are not chemically bonded to the cork surface, resulting in a reversible phenomenon (Lequin et al., 2010). On the contrary, the ethanol sorption phenomenon occurs following a chemisorption mechanism, resulting in an irreversible phenomenon (Lequin et al., 2013). The presence of ethanol during the storage period led to a reduction in the stiffness of cork, highlighting a significant potential impact for natural cork stoppers for oenological applications.

3.2. Effect of ethanol and water vapour sorption on the mechanical response of cork to PRL cycles

An example of the mechanical response to the PRL test of t_3 cork cubes placed in contact with ethanol vapour is illustrated in Fig. 3a. It is noticeable that, from the 1st to the 5th cycle, repetitions *b* and *c* are closer together than *a*. Moreover, when the next cycle was performed at a higher strain level, repetitions *b* and *c* of the previous cycle almost overlapped with the repetition *a* of the next cycle (Fig. 3a). Furthermore, the *a, b* and *c* repetitions of the 1st cycle as well as the *a* repetition of the

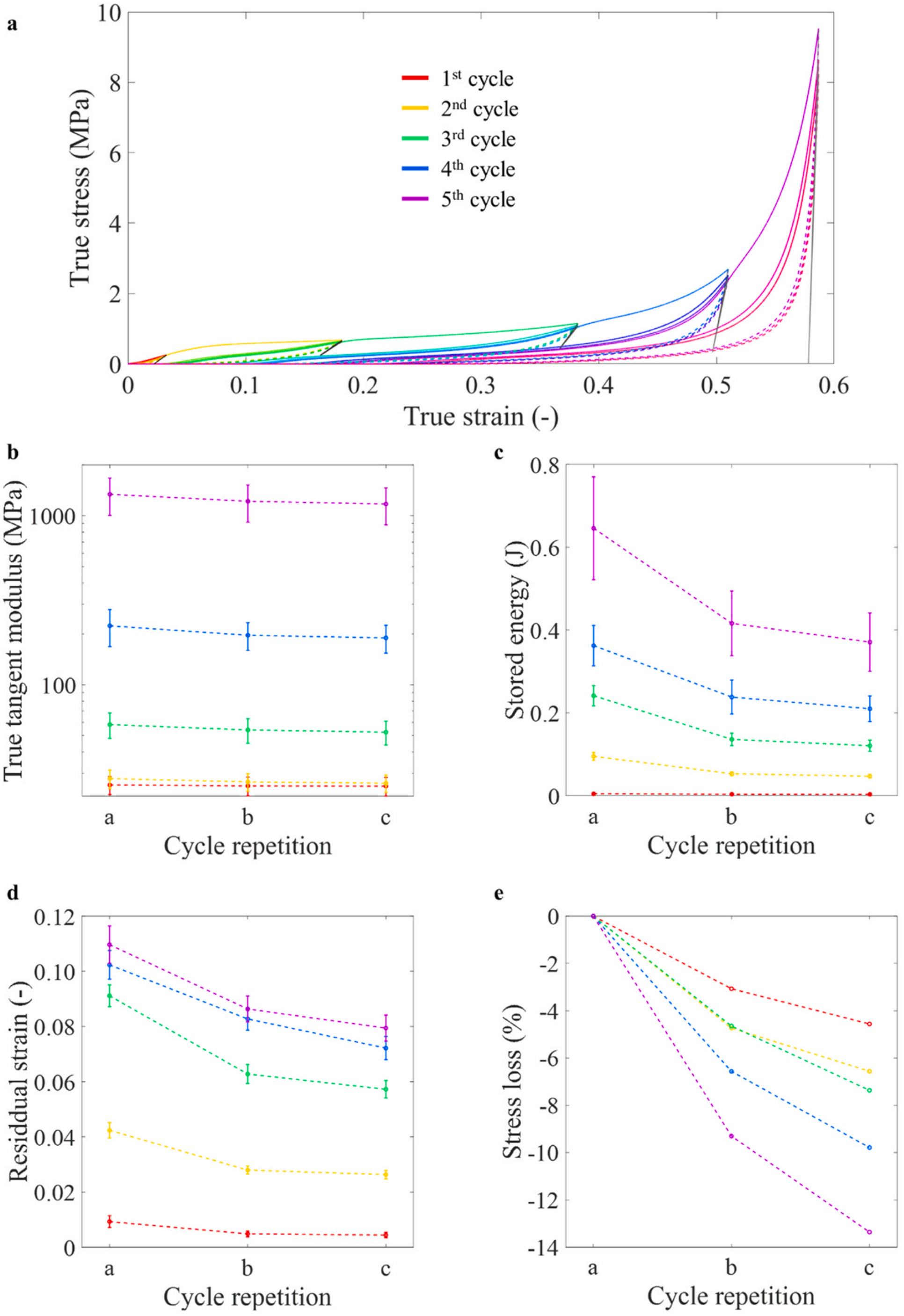

Fig. 3. (a) True stress-strain curves obtained for the repetition of 5 consecutive progressive cycles performed on cork cubes previously stored for 3 months in contact with ethanol vapour at 25 °C (n = 4). (b) Tangent modulus E_t (MPa) as a function of cycle repetitions. (c) Stored energy W (J) as a function of cycle repetitions. (d) Residual strain ε_r (-) as a function of cycle repetitions (e) Stress loss σ_l (%) as a function of cycle repetitions (for Fig. 3b, c, d, e dashed line is a guide to the eyes).

2nd cycle were characterised by an initial single linear slope, while from the *b* repetition of the 2nd cycle to the end of the 5th cycle, a variation in the slope was observed. When cork was compressed up to 18 % of strain (2nd cycle), *a, b* and *c* repetitions of the 1st cycle overlapped with the *a* repetition of the 2nd cycle. Then, from *b* repetition of the 2nd cycle, the loading curves did not overlap, and a stress loss appeared. It is also worthy to note that, between the loading and unloading curve a hysteresis phenomenon was observed. Although it was very small for the 1st cycle, it became more important from the 2nd to the 5th cycle. The corresponding stress-strain curves performed by PRL test for cork samples placed in contact with water, water-ethanol (both for t_3 and t_{12}) and ethanol (t_{12}) are presented as appendices, in figures F.7, G.8, H.9, I.10 and L.11, respectively.

3.2.1. *Tangent modulus (E_t)*

Fig. 3b shows the typical evolution of the tangent modulus as a function of the unloading cycle repetitions (*a, b, c*) for the five cycles for t_3 cork cubes placed in contact with ethanol vapour. From the 1st (3 % of strain) to the 5th cycle (58 % of strain), the tangent modulus increased from 25.5 MPa to 1339.0 MPa, resulting in an overall stiffening of the material at discharge. Similarly, it increased from 17.3 MPa to 1093.1 MPa for water vapor and from 17.9 MPa to 1216.7 MPa for water-ethanol vapour (Figures F.7b and G.8b shown in appendices). This phenomenon was due to the progressive buckling of the cell walls following the application of strain levels from 3 % (1st cycle) to 58 % (5th cycle). In particular, the results obtained for samples placed in contact with water vapour were similar to those obtained in a previous study by carrying out five PRL cycles (but without repetition) on cork cubes stored at 25 °C and ~50 % RH (Gerometta et al., 2023). The tangent modulus obtained in the present work decreased of about 50 % compared to the values obtained for cork cubes stored at ~50 % RH. As already discussed for the QS test, this reduction is attributed to the formation of water clusters in the cork polymer chains and plasticization of the material (Lagorce-Tachon et al., 2015).

Another important aspect to consider concerns the decrease of the tangent modulus following repetitions performed within a given cycle. It slightly decreased of about 1.5 % between *a* and *c* repetitions of the 1st cycle. This reduction was amplified to 6.2 % for the 2nd cycle, 9.7 % for the 3rd cycle, 15.2 % for the 4th cycle and 12.4 % for the 5th cycle. Similarly to the trend observed for ethanol, a reduction in tangent modulus values between *a* and *c* repetitions of about 0.9 %, 1.1 %, 3.3 %, 6.2 %, 8.8 % (from 1st to 5th cycle) was also observed for cork cubes stored in water vapour condition at t_3 (F.7b). For samples placed in contact with water-ethanol vapour, except for the 1st cycle for which no change in the tangent modulus was observed, it decreased by 1.3 % (2nd cycle), 4.9 % (3rd cycle), 8.7 % (4th cycle) and 10.1 % (5th cycle) (Fig. G.8b). It is therefore interesting to note that, regardless to the storage condition, there is evidence of an important contribution of the inelastic component of the material to the mechanical behaviour of cork, as a part of the material's deformation remains non-recoverable after the applied load is removed over the repeated cycles. The tangent modulus was not significantly affected by the storage condition. At t_3, only the first two cycles for ethanol vapour were significantly different from the other two storage conditions (Table A.1). At t_{12}, a significant difference was observed for the 1st, 2nd, and 3rd cycles, between samples placed in contact with water-ethanol and water vapours. No difference was noted for ethanol and water vapours storage conditions (Table A.1).

From the perspective of mechanical properties, the tangent modulus calculated from the PRL test as well as the elastic modulus obtained from the QS test, can be used to characterise the stiffness of the material. However, they provide limited information on its compressive behaviour for a given strain level. Compared to the QS test, the PRL test allows a more in-depth analysis of the changes in the mechanical behaviour of cork in the plateau and densification regions, by providing further information on stored energy, stress loss and residual strain.

3.2.2. *Stored energy (W)*

This parameter represents the energy absorbed by cork during the compression cycle. The stored energy as a function of the cycle repetitions *a, b* and *c* is shown in Fig. 3c for cork cubes (t_3) placed in contact with ethanol vapour. The stored energy increased throughout the five cycles from 4.5 mJ to 645.9 mJ. This highlights that cork is a material which absorbs more energy when subjected to higher compression levels, confirming its excellent ability to withstand high compression, as already proven by the previous QS test. However, the stored energy was affected by the repetitions (*a, b, c*) within each cycle. A reduction of 27 % (from 4.5 mJ to 3.3 mJ) for the 1st cycle was determined. Then, the reduction reached 50 % following the application of 2nd (from 95.1 mJ to 47.1 mJ) and 3rd (from 241.4 mJ to 120.7 mJ) cycles. Finally, the decrease in stored energy was 43 % for the 4th (362.4 mJ to 209.9 mJ), and 5th (645.9 mJ to 370.9 mJ) cycles.

Considering these results, although cork maintained a relatively high good stiffness at discharge (high value of the tangent modulus), it absorbed less energy following the repetition of each cycle. This highlights that its compressive behaviour was modified during the PRL test. For each of the five cycles, the highest reduction occurred between *a* and *b* repetitions: ~22 % for the 1st cycle, ~44 % for the 2nd and 3rd cycles and ~36 % for the 4th cycle and 5th cycles. This phenomenon is clearly observed from the 2nd to the 5th cycles, whereas it is less pronounced in the case of the 1st cycle (Fig. 3c). The same trend was also observed for cork stored with water and water-ethanol vapours (Figures in appendices F.7c, G.8c). No significant difference was noticeable between storage conditions at t_3, except for the *a* repetition of the 2nd cycle, when cork was exposed to ethanol compared to water and water-ethanol vapours (Table B.2). At t_{12}, a significant difference was observed only for the 1st cycle, between ethanol or water-ethanol and water vapour storage conditions (Table B.2).

As cork is a cellular material with a honeycomb structure (Crouvisier-Urion et al., 2019), its stress-strain curve is similar to that of polymer foams which are also characterised by an elastic, a plateau and a densification regions (Rahimidehgolan and Altenhof, 2023). Thus, the mechanical response of cork following PRL cycles is also similar to that of polymer foams (Xia et al., 2009; Peroni and Peroni, 2009; Ozturk and Anlas, 2009; Shen and Golnaraghi, 2001). Considering polypropylene foam, Yang *et al. (*Yang et al., 2011*)* carried out an uniaxial PRL cycles at 25 %, 55 % and 90 % of strain at a crosshead displacement rate of 10 mm·min^{-1}. The PRL test was performed with repeated cycles at different time intervals of 1 min and 1 month. For both tests, the polypropylene foam showed a significant reduction in energy absorption capability following the application of repeated cycles. This phenomenon is known as stress softening, and it is due to the damage of the foam's cell walls. It implies that a lower force is required to compress the material. It depends on the deformation history of the material. The maximum deformation applied during a cycle act as a threshold. Until this maximum deformation is not exceeded, (before starting the new cycle), the mechanical behaviour of foam shows a stress-strain loading curve with stress softening. When this threshold is exceeded by performing a new cycle at a higher strain level, the stress-strain loading curve of the new cycle approached the last stress-strain loading curve of the previous cycle without any softening. This phenomenon then reappears for subsequent repetitions of the new cycle. The unloading curves of each cycle repetitions are not affected by stress softening (Yang et al., 2011). The mechanical response of cork to PRL cycles showed a similar behaviour. While no change in slope was noted between repetitions of the 1st cycle, from the *a* repetition of the 2nd to the *c* repetition of 5th cycle, an important stress softening clearly appeared, as highlighted by the change in slopes on the loading curves (Fig. 3a). This led to hypothesis that a structural damage in the cork structure occurred after the application of compression cycles exceeding the elastic region of the material, with the appearance of residual strain and stress loss phenomena. However, SEM analysis did not reveal any structural damage at the cellular level between the compressed and uncompressed samples.

No delamination of the cell wall occurred, even after the compression test (appendices O, P, and Q). To go further *in situ* test coupling compression and SEM observations could be helpful in better understanding this phenomenon.

3.2.3. Residual strain (ε_r)

The residual strain as a function of the repetitions *a, b* and *c* of the five cycles for cork samples stored in contact with ethanol vapour are displayed in Fig. 3d. It increased from 0.009 to 0.11 considering the *a* repetitions from the 1st to the 5th cycle. However, a reduction in residual strain was observed between the application of *a* and *b* repetitions of about 48 % for the 1st cycle, 34 % for the 2nd cycle, 31 % for the 3rd cycle, 19 % for the 4th cycle and 21 % for the 5th cycle. An additional slight decrease between 4 % and 10 % was observed between of *b* and *c* repetitions, for each of the five cycles. A similar trend was noted for samples in contact with water and water-ethanol vapours. No significance effect of the storage condition was highlighted for the residual strain at t_3 and t_{12}, except for the 2nd and 3rd cycles for samples stored with water-ethanol vapour that were significantly different from those exposed to pure substances (water or ethanol) (Table C.3).

3.2.4. Stress loss (σ_l)

The stress loss as a function of the repetitions *a, b* and *c* for each of the five cycles for cork samples stored in contact with ethanol vapour is shown in Fig. 3e. As already observed for the other parameters, stress loss decreased between cycle repetitions, assuming that damage in the cork structure occurred when cork was subjected to successive compression cycles. A reduction of ~4 % for the 1st cycle (~0.3 MPa) was observed. Then, stress loss value reached ~7 % after the 2nd and 3rd cycles (~0.7 MPa and ~3.8 MPa), ~10 % after the 4th cycles (~3 MPa) and ~13 % after the 5th cycle (~11 MPa). No significant difference was noticeable between storage conditions at t_3, except for the 2nd cycle and for *a* repetition of the 3rd cycle, between pure substances (ethanol and water) (Table D.4). At t_{12}, a significant difference was observed for the 1st cycle, between samples placed in contact with ethanol or water-ethanol vapours and those exposed to water vapour. The 3rd cycle corresponding to water and water-ethanol storage conditions were also different (Table D.4).

These results clearly show that inelastic phenomena in the cork structure occurred from the 2nd (18 % true deformation) to the 5th (58 % true deformation) cycle. Indeed, from a mechanical point of view, cork is considered as a time-dependant (viscoelastic) material (Mano, 2002). When a small deformation is applied, it exhibits an elastic behaviour. It thus recovers its original shape once the stress is removed. When subjected to high deformation, viscous behaviour starts to appear. In this case, cork does not fully recover its shape when the stress is removed (Mano, 2002). From the PRL test, the overlapping of the *a, b* and *c* repetitions of the 1st cycle with the *a* repetition of the 2nd cycle corresponds to the elastic region of the stress-strain curve of cork, in which only reversible modifications in the cork structure occur. The very small hysteresis and stress loss phenomena observed for *a*, *b*, and *c* repetitions of the 1st cycle may be attributed to the viscous component of the cell walls. This component may be characterised by multiple relaxation times, ranging from instantaneous to extremely slow recovery, resulting in delayed changes in the structure of the material.

For deformation exceeding the elastic region (3 % true strain), an important hysteresis, stress loss, and residual strain phenomena appear on the stress-strain curve (Fig. 3c, 3d, 3e). This means that irreversible structural damage in the cell walls can occur. This phenomenon can be observed by the variation in the slope of the loading curve from *b* repetition of the 2nd cycle (18 % true strain) to the 5th cycle (58 % true strain) (Fig. 3a). In a previous study, Rosa and Fortes analysed the dimension recovery of 16 mm edge cork cubes after performing a quasi-static compression test. After the application of 30 % nominal strain, they observed an almost full dimension recovery (less than 1 % of residual deformation in the compression direction) at 20 °C after 20 days. However, the test performed at 80 % nominal strain induced a permanent deformation of the cork cube, remaining even after 70 days (more than 5 % of residual deformation in the compression direction) (Rosa and Fortes, 1988a). The same authors also investigated the stress relaxation and creep behaviour of cork by applying a compression test with loading-relaxation-unloading cycles to simulate the performance of a cork stopper (30 % of strain compression in the tangential direction, 10 min relaxation, 10 min in unloaded condition during 10 cycles). The stress relaxation phenomenon associated with the unfolding process of the buckled cell walls was assumed to be responsible for the lack of dimensional recovery of cork (Rosa and Fortes, 1988a). Suberin was hypothesized to be the macromolecule responsible for this phenomenon, as reported in a more recent study about the viscoelastic properties of cork (Mano, 2002).

In summary, although the impact of the storage condition at t_3 and t_{12} was not clearly demonstrated for the PRL parameters, a potential effect of ethanol vapour was noticeable during storage time between 3 (t_3) and 12 (t_{12}) months. Among the PRL parameters, the stored energy was selected as the most appropriate for studying the evolution of the mechanical behaviour of cork over time.

3.3. Effect of storage time on the compressive behaviour of cork subjected to successive cycles

The effect of storage time on the compressive behaviour of cork was also studied in the present work by performing a PRL test to characterise changes in the mechanical behaviour of cork which may occur in the bottleneck of the wine bottle (mimicking especially vertical storage). The stored energy values obtained for cork placed in contact with water (W), water-ethanol (W-E) and ethanol (E) vapours, at t_3 and t_{12}, are displayed in Fig. 4a and b, respectively. For each storage condition, the blue, orange and yellow bars correspond to the *a, b* and *c* repetitions of each of the five PRL cycles.

It is worthy to note that at t_3, for the *a* repetition from the 2nd to the 5th cycle, cork cubes exposed to ethanol vapour showed slightly higher stored energy values than those determined for water-ethanol vapour and rather high values compared to water vapour ($W_{a,\ ethanol} > W_{a,\ water\text{-}ethanol} > W_{a,\ water}$). After 12 months of storage the ranking is reversed: the highest values were observed for cork cubes in contact with water vapour, followed by those of samples exposed to water-ethanol and ethanol vapours, characterised by similar values of stored energy. This phenomenon was less pronounced for *b* and *c* cycle repetitions at t_3 and t_{12}. Although no significant effect of storage condition was evidenced at t_3 and at t_{12} (Table B.2), it is interesting to assess the effect of time on the mechanical behaviour of cork for the same storage condition. There was no significant difference of stored energy values between t_3 and t_{12} for cork cubes stored in contact with water vapour. In the case of water-ethanol vapour, although the 1st cycle was not different, the 2nd, 3rd, 4th and 5th cycles were different between t_3 and t_{12}. In the case of ethanol vapour, the effect was even more pronounced, showing a significant difference in stored energy values for all PRL cycles between t_3 and t_{12} (Table B.2). Similarly, the effect of time was also noticeable for other PRL parameters. Although no difference in the tangent modulus and stress loss values between t_3 and t_{12} was observed during water vapour storage (Table A.1, D.4), a significant difference for samples in contact with water-ethanol vapour was noted for the tangent modulus (1st, 2nd and 3rd cycles were different, Table A.1) and stress loss (2nd, 3rd, and 4th cycles were different, Table D.4). In the case of cork stored in contact with ethanol vapour, these parameters were different for all cycles (Tables A.1, D.4). To highlight the effect of storage time on the compressive behaviour of cork, the relative variation of stored energy ΔW (%) was calculated as follows:

$$\Delta W = \frac{W_{12} - W_3}{W_3} \times 100 \qquad (1)$$

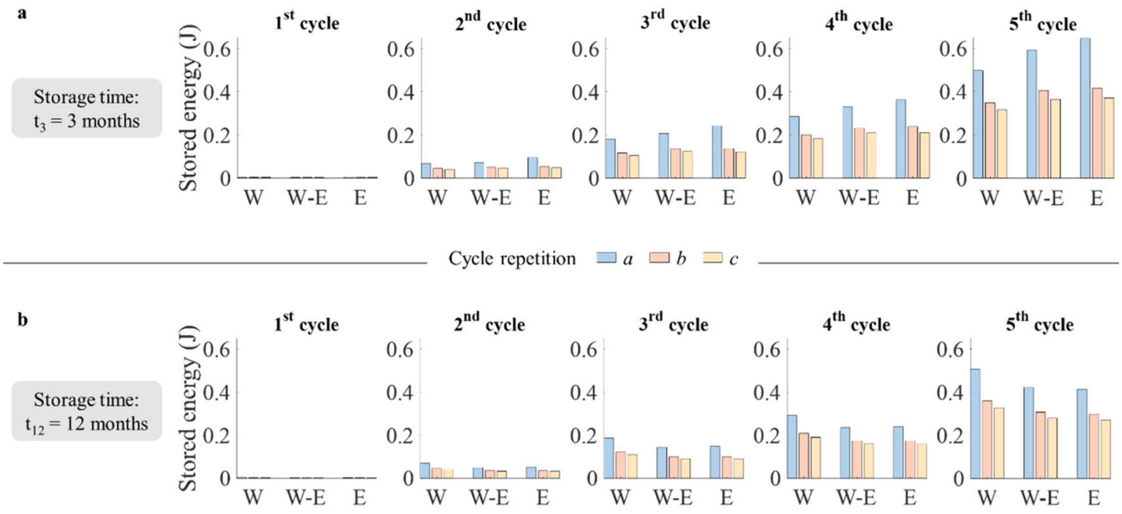

Fig. 4. Stored energy (W) following the application of five successive compression cycles (PRL test) carried out on cork cubes (a) after 3 months and (b) 12 months of storage in contact with water, water-ethanol or ethanol vapours.

With W_{12} – W_3 *(J)* is the difference between stored energy values obtained for each cycle repetition at t_{12} and t_3, normalised by W_3 *(J)*.

The relative variation of stored energy (ΔW) for the five PRL cycles performed on cork stored under different storage conditions is displayed in Fig. 5. It unambiguously shows that, after 12 months of storage, cork did not have the same capability to store energy. Interestingly, ΔW increased for samples in contact with water vapour (positive values). In this case, following the first repetition of each cycle, ΔW of *b* and *c* repetitions increased of $\sim$5 % (1st cycle) and $\sim$2 % (2nd to 5th cycles), respectively. When cork was exposed to water-ethanol or ethanol vapours its capability to store energy was significantly reduced (negatives values). Contrary to what observed for water vapour, for *b* and *c* repetitions a reduction of |ΔW| $\sim$2 % for the 1st cycle up to $\sim$5 % for the 5th cycle was noted for sample in contact with water-ethanol vapour. Similarly, that decrease in |ΔW| reached $\sim$10 % following the 5th cycle when cork was placed under ethanol vapour. It is also worthy to note that the mechanical response of cork over the cycles was different in relation to the storage condition. When cork was exposed to water vapour, a reduction in ΔW by a factor 3 was observed after the 1st cycle (3 % of strain). Then, between the 2nd and 5th cycle, this value remained stable at $\sim$5 %. In contrast, in the case of water-ethanol and ethanol vapours a progressive decrease in |ΔW| was noted from the 1st to the 5th cycle by a factor 1.3 and 1.7, respectively. These results revealed that the storage condition, particularly the presence of ethanol vapour, affects the ageing of the material and has an important effect on the compressive behaviour of cork.

It is well known that cork has a certain affinity for water (Chanut et al., 2022). Water is sorbed on cork following a physisorption mechanism. Firstly, the hydroxyl and methoxyl groups act as localised hydrophilic sites where hydrogen bonds can easily be established with water molecules. Secondly, the adsorption phenomenon continues by the formation of water clusters around these hydrophilic sites. When cork is placed in contact with water vapour at high relative humidity close to saturation, the adsorbed amount of water is $\sim$5 mmol.g^{-1}. The adsorption-desorption phenomenon is a reversible process, thus no water remains chemically bonded to the cork surface after vacuum treatment (Lequin et al., 2010). Cork has also affinity with ethanol. In this case chemisorption occurs in addition to physisorption. At the

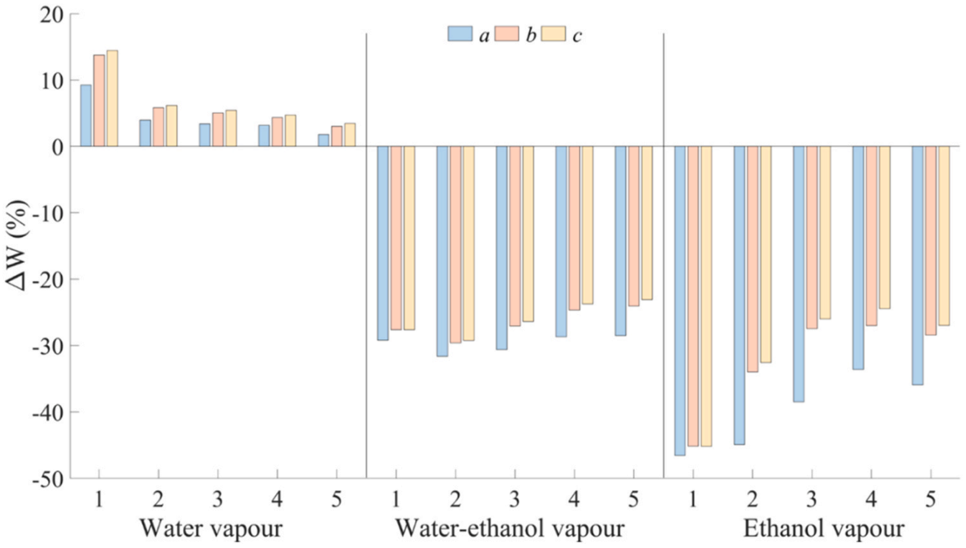

Fig. 5. Relative variation of the stored energy as a function of the 5 successive compression cycles (from PRL test) determined for cork cubes stored from 3 to 12 months in contact with water, water-ethanol and ethanol vapours. The blue, Orange and yellow bars represent the first "*a*", second "*b*" and third "*c*" repetition of each cycle, respectively (n = 4).

relative pressure of 0.7 the sorbed amount of ethanol is ~1 mmol.g^{-1} and the adsorption-desorption phenomenon is irreversible. In this case desorption is not complete, and the amount of ethanol that remained chemically bonded to the cork surface is about ~0.25 mmol.g^{-1} (Lequin et al., 2013). The cork cells wall is composed of suberin (~40 % wt.), lignin (~22 % wt.) and polysaccharides (cellulose and hemicelluloses, ~18 % wt) (Silva et al., 2005). Ethanol is a solvent used for lignin and suberin extraction (Cordeiro et al., 2002). It can be assumed that the reaction between ethanol and these components leads to a structural change in cork over time, resulting in a different compressive behaviour over 1-year storage. A relatively recent study reported that ethanol in the supercritical state can lead to significant liquefaction of the lignin fraction, as revealed by FTIR analysis (Bui et al., 2015). Indeed, they found that the solid recovered after treatment of biomass with supercritical ethanol at 280 °C showed changes in the relative intensities of some characteristic bands at 1720 cm^{-1} (νC=O of carboxylic groups) and at 1514 cm^{-1} (νC=C of aromatic ring). The absorption was significantly lower compared to initial untreated lignin, suggesting chemical reactivity between these two components (Bui et al., 2015).

4. Conclusions

This study investigated the effect of the environmental condition of storage on the compressive behaviour of natural cork over time. The focus on the role of ethanol is especially relevant for applications in alcoholic beverage preservation.

From quasi-static compression tests:

- At 3 months of storage, samples in contact with ethanol exhibited higher stiffness compared to those exposed to water or water-ethanol vapours.
- At 12 months of storage, a significant decrease in elastic modulus, and consequently in stiffness, was observed for cork exposed to ethanol vapour. However, no significant changes were noted for cork stored in contact with water or water-ethanol vapour.

From progressive repeated loading tests:

- No significant effect of storage conditions was observed on the tangent modulus, stored energy, residual strain and stress loss at either t_3 or at t_{12}.
- Notwithstanding the storage condition, cork demonstrated remarkable resilience, maintaining approximately 50 % of its energy storage capacity even after undergoing PRL cycles, highlighting its exceptional compressibility.
- In ethanol vapour condition, the energy storage capacity decreased by ~40 % between 3 and 12 months.
- Under water storage condition, the energy storage capacity increased by ~15 % over the same period.

These results suggest that ethanol plays a key role in altering the mechanical behaviour of cork, particularly in scenarios such as vertical storage of alcoholic beverages, where the cork is in direct contact with ethanol vapours. Mechanical ageing of cork stoppers is of particular importance in the field of oenology and wine bottle ageing, especially since there is currently no reliable mechanical characterization method that accurately reflects the ageing state of the material under various environmental conditions of storage. The mechanical changes observed could influence the long-term performance of the cork–bottleneck system, particularly its barrier properties. In the present study, a novel mechanical characterization approach was applied to cork material to identify indicators capable of effectively capturing the evolution of materialist properties over time.

The results also support the hypothesis that ethanol accelerates mechanical ageing, likely due to its chemical reactivity with lignin and suberin. The macroscopic effect of ethanol in cork stiffness observed during ageing highlights the need for in-depth study on a molecular scale to better understand the mechanisms of ethanol reactivity with cork components. Furthermore, similar investigations should be extended to cork exposed to water vapour, in order to explain the different mechanical behaviour observed during ageing compared to ethanol exposure.

To go further, the effect of direct liquid contact with different water-ethanol solutions on the compressive behaviour of cork will be worthwhile to study. In addition, a deeper understanding of the chemical reactivity between ethanol and cork will be essential to fully elucidate the impact of ethanol on cork structure and performance.

CRediT authorship contribution statement

Thomas Karbowiak: Writing – review & editing, Validation, Software, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Massimiliano Gerometta:** Writing – original draft, Validation, Software, Methodology, Investigation, Formal analysis, Conceptualization. **Sébastien Thibaud:** Writing – review & editing, Validation, Software, Methodology, Investigation, Formal analysis, Conceptualization. **François Villette:** Writing – review & editing, Validation, Investigation, Formal analysis. **Aurélie Lagorce:** Writing – review & editing, Validation, Software, Methodology, Investigation, Formal analysis, Conceptualization. **Xavier Gabrion:** Writing – review & editing, Validation, Software, Methodology, Investigation, Formal analysis, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.indcrop.2025.121333.

Data availability

Data will be made available on request.

References

AFNOR, 1995. Bottling industry. Glass bottles. Glass Crown Champagne Finish 26, 29.

Amorim. 2024; Available from: https://www.amorimcork.com/en/cork-and-wine/raw-material-and-production-process/.

Anjos, O., Pereira, H., Rosa, M.E., 2008. Effect of quality, porosity and density on the compression properties of cork. Holz als Roh und Werkst. 66 (4), 295–301. https://doi.org/10.1007/s00107-008-0248-2.

Anjos, O., Pereira, H., Rosa, M.E., 2010. Tensile properties of cork in the tangential direction: variation with quality, porosity, density and radial position in the cork plank. Mater. Des. 31 (4), 2085–2090. https://doi.org/10.1016/j.matdes.2009.10.048.

Anjos, O., Rodrigues, C., Morais, J., Pereira, H., 2014. Effect of density on the compression behaviour of cork. Mater. Des. 53, 1089–1096. https://doi.org/10.1016/j.matdes.2013.07.038.

APCOR, 2020. Cork Yearbook. Associação Portuguesa da Cortiça: Av. Comendador Henrique Amorim, n.º 580, Apartado 100, 4536-904 Santa Maria de Lamas, Portugal.

Bui, N.Q., Fongarland, P., Rataboul, F., Dartiguelongue, C., Charon, N., Vallée, C., Essayem, N., 2015. FTIR as a simple tool to quantify unconverted lignin from chars in biomass liquefaction process: application to SC ethanol liquefaction of pine wood. Fuel Process. Technol. 134, 378–386. https://doi.org/10.1016/j.fuproc.2015.02.020.

Chanut, J., Bellat, J.-P., Gougeon, R.D., Karbowiak, T., 2023. A key to wine conservation lies in the glass/cork interface. PNAS Nexus. https://doi.org/10.1093/pnasnexus/pgad344.

Chanut, J., Wang, Y., Dal Cin, I., Ferret, E., Gougeon, R.D., Bellat, J.P., Karbowiak, T., 2022. Surface properties of cork: is cork a hydrophobic material? J. Colloid Interface Sci. 608, 416–423. https://doi.org/10.1016/j.jcis.2021.09.140.

Charte des bouchonniers liègeurs, 2006.

Coelho, R.M., Alves de Sousa, R.J., Fernandes, F.A.O., Teixeira-Dias, F., 2013. New composite liners for energy absorption purposes. Mater. Des. 43, 384–392. https://doi.org/10.1016/j.matdes.2012.07.020.

Cordeiro, N., Neto, C.P., Rocha, J., Belgacem, M.N., Gandini, A., 2002. The organosolv fractionation of cork components. Holzforschung 56 (2), 135–142. https://doi.org/10.1515/HF.2002.023.

Crouvisier-Urion, K., Bellat, J.-P., Gougeon, R.D., Karbowiak, T., 2018a. Gas transfer through wine closures: a critical review. Trends Food Sci. Technol. 78, 255–269. https://doi.org/10.1016/j.tifs.2018.05.021.

Crouvisier-Urion, K., Bellat, J.-P., Gougeon, R.D., Karbowiak, T., 2018b. Mechanical properties of agglomerated cork stoppers for sparkling wines: influence of adhesive and cork particle size. Compos. Struct. 203, 789–796. https://doi.org/10.1016/j.compstruct.2018.06.116.

Crouvisier-Urion, K., Chanut, J., Lagorce, A., Winckler, P., Wang, Z., Verboven, P., Nicolai, B., Lherminier, J., Ferret, E., Gougeon, R.D., Bellat, J.P., Karbowiak, T., 2019. Four hundred years of cork imaging: new advances in the characterization of the cork structure. Sci. Rep. 9 (1), 19682. https://doi.org/10.1038/s41598-019-55193-9.

Dias, S., Tadeu, A., António, J., Almeida, J., Pedro, F., Martins, S., Serra, C., 2018. Experimental study of expanded cork agglomerate blocks – compressive creep behavior and dynamic performance. Constr. Build. Mater. 181, 551–564. https://doi.org/10.1016/j.conbuildmat.2018.06.021.

Echave, J., Barral, M., Fraga-Corral, M., Prieto, M.A., Simal-Gandara, J., 2021. Bottle aging and storage of wines: a review. Molecules 26 (3), 713. https://doi.org/10.3390/molecules26030713.

Ferreira Serra, G., Fernandes, F.A.O., J. Alves de Sousa, R., Noronha, E., Ptak, M., 2022a. New hybrid cork-STF (Shear thickening fluid) polymeric composites to enhance head safety in micro-mobility accidents. Compos. Struct. 301, 116138. https://doi.org/10.1016/j.compstruct.2022.116138.

Ferreira Serra, G., Fernandes, F.A.O., J. Alves de Sousa, R., Noronha, E., Ptak, M., 2022b. New hybrid cork-STF (Shear thickening fluid) polymeric composites to enhance head safety in micro-mobility accidents. Compos. Struct. 301, 116–138. https://doi.org/10.1016/j.compstruct.2022.116138.

Flores, M., Rosa, M.E., Barlow, C.Y., Fortes, M.A., Ashby, M.F., 1992. Properties and uses of consolidated cork dust. J. Mater. Sci. 27 (20), 5629–5634. https://doi.org/10.1007/BF00541634.

Fonseca, A.L., Brazinha, C., Pereira, H., Crespo, J.G., Teodoro, O.M.N.D., 2013. Permeability of cork for water and ethanol. J. Agric. Food Chem. 61 (40), 9672–9679. https://doi.org/10.1021/jf4015729.

Fortes, M.A., Teresa Nogueira, M., 1989. The poisson effect in cork. Mater. Sci. Eng. A 122 (2), 227–232. https://doi.org/10.1016/0921-5093(89)90634-5.

Gerometta, M., Gabrion, X., Lagorce, A., Thibaud, S., Karbowiak, T., 2023. Towards better understanding of the strain–stress curve of cork: a structure–mechanical properties approach. Mater. Des. 235. https://doi.org/10.1016/j.matdes.2023.112376.

Gibson, L.J. and Ashby, M.F., Cellular Solids: Structure and Properties, 2 ed. Cambridge University Press: Cambridge, 1997.

Gibson, L.J., Easterling, K.E., Ashby, M.F., 1981. The structure and mechanics of cork. Proc. R. Soc. Lond. A. Math. Phys. Sci. 377 (1769), 99–117. https://doi.org/10.1098/rspa.1981.0117.

IOVW, 2024. International Code Of Oenological Practices. International Organisation of Vine and Wine. URL https://www.oiv.int/standards/international-code-of-oenological-practices.

Kaczyński, P., Ptak, M., A. O. Fernandes, F., Chybowski, L., Wilhelm, J., J. Alves de Sousa, R., 2019. Development and testing of advanced cork composite sandwiches for energy-absorbing structures. Materials 12 (5), 697. https://doi.org/10.3390/ma12050697.

Karbowiak, T., Crouvisier-Urion, K., Lagorce, A., Ballester, J., Geoffroy, A., Roullier-Gall, C., Chanut, J., Gougeon, R.D., Schmitt-Kopplin, P., Bellat, J.P., 2019. Wine aging: a bottleneck story. NPJ Sci. Food 3, 14. https://doi.org/10.1038/s41538-019-0045-9.

Lagorce-Tachon, A., Karbowiak, T., Champion, D., Gougeon, R.D., Bellat, J.-P., 2015. Mechanical properties of cork: effect of hydration. Mater. Des. 82, 148–154. https://doi.org/10.1016/j.matdes.2015.05.034.

Lagorce-Tachon, A., Karbowiak, T., Champion, D., Gougeon, R.D., Bellat, J.-P., 2016. How does hydration affect the mechanical properties of wine stoppers? J. Mater. Sci. 51 (9), 4227–4237. https://doi.org/10.1007/s10853-015-9669-6.

Lakreb, N., Şen, U., Toussaint, E., Amziane, S., Djakab, E., Pereira, H., 2023. Physical properties and thermal conductivity of cork-based sandwich panels for building insulation. Constr. Build. Mater. 368, 130–420. https://doi.org/10.1016/j.conbuildmat.2023.130420.

Lequin, S., Chassagne, D., Karbowiak, T., Gougeon, R., Brachais, L., Bellat, J.P., 2010. Adsorption equilibria of water vapor on cork. J. Agric. Food Chem. 58 (6), 3438–3445. https://doi.org/10.1021/jf9039364.

Lequin, S., Chassagne, D., Karbowiak, T., Bellat, J.P., 2013. Sorption equilibria of ethanol on cork. J. Agric. Food Chem. 61 (22), 5391–5396. https://doi.org/10.1021/jf4016043.

Lopes, P., Saucier, C., Teissedre, P.-L., Glories, Y., 2006. Impact of storage position on oxygen ingress through different closures into wine bottles. J. Agric. Food Chem. 54 (18), 6741–6746. https://doi.org/10.1021/jf0614239.

Lopes, P., Saucier, C., Teissedre, P.-L., Glories, Y., 2007. Main routes of oxygen ingress through different closures into wine bottles. J. Agric. Food Chem. 55 (13), 5167–5170. https://doi.org/10.1021/jf0706023.

Maga, J.A., Puech, J.-L., 2005. Cork and alcoholic beverages. Food Rev. Int. 21 (1), 53–68. https://doi.org/10.1081/FRI-200040593.

Mano, J.F., 2002. The viscoelastic properties of cork. J. Mater. Sci. 37 (2), 257–263. https://doi.org/10.1023/A:1013635809035.

Oliveira, V., Rosa, M.E., Pereira, H., 2014. Variability of the compression properties of cork. Wood Sci. Technol. 48 (5), 937–948. https://doi.org/10.1007/s00226-014-0651-2.

Ozturk, U.E., Anlas, G., 2009. Energy absorption calculations in multiple compressive loading of polymeric foams. Mater. Des. 30 (1), 15–22. https://doi.org/10.1016/j.matdes.2008.04.054.

Pereira, H., 2007. Chapter 11 - Cork products and uses. In: Pereira, H. (Ed.), Cork. lsevier Science B.V., Amsterdam, pp. 243–261. https://doi.org/10.1016/B978-044452967-1/50013-3.

Pereira, H., Graça, J., Baptista, C., 1992. The effect of growth rate on the structure and compressive properties of cork. IAWA J. 13 (4), 389–396. https://doi.org/10.1163/22941932-90001294.

Peroni, L., Peroni, A.M., 2009. M., the mechanical behaviour of polyurethane foam: multiaxial and dynamic behaviour. Int. J. Mater. Eng. Innov. 1 (2), 154–174. https://doi.org/10.1504/IJMatEI.2009.029362.

Rahimidehgolan, F., Altenhof, W., 2023. Compressive behavior and deformation mechanisms of rigid polymeric foams: a review. Compos. Part B Eng. 110–513. https://doi.org/10.1016/j.compositesb.2023.110513.

Reculusa, S., Trinquecoste, M., Dariol, L., Delhaès, P., 2006. Formation of low-density carbon materials through thermal degradation of a cork-based composite. Carbon 44, 1316–1320. https://doi.org/10.1016/j.carbon.2005.12.051.

Rosa, M.E., Fortes, M.A., 1988a. Stress relaxation and creep of cork. J. Mater. Sci. 23 (1), 35–42. https://doi.org/10.1007/BF01174031.

Rosa, M.E., Fortes, M.A., 1988a. Rate effects on the compression and recovery of dimensions of cork. J. Mater. Sci. 23 (3), 879–885. https://doi.org/10.1007/BF01153983.

Rosa, M.E., Fortes, M.A., 1988b. Temperature-induced alterations of the structure and mechanical properties of cork. Mater. Sci. Eng. 100, 69–78. https://doi.org/10.1016/0025-5416(88)90240-6.

Sarasini, F., Tirillò, J., Lampani, L., Sasso, M., Mancini, E., Burgstaller, C., Calzolari, A., 2019. Static and dynamic characterization of agglomerated cork and related sandwich structures. Compos. Struct. 212, 439–451. https://doi.org/10.1016/j.compstruct.2019.01.054.

Shen, Y., Golnaraghi, F., 2001. And plumtree, A., modelling compressive cyclic stress–strain behaviour of structural foam. Int. J. Fatigue 23 (6), 491–497. https://doi.org/10.1016/S0142-1123(01)00014-7.

Silva, S.P., Sabino, M.A., Fernandes, E.M., Correlo, V.M., Boesel, L.F., Reis, R.L., 2005. Cork: properties, capabilities and applications. Int. Mater. Rev. 50 (6), 345–365. https://doi.org/10.1179/174328005X41168.

Suhas, E., Thibon, C., Loisel, C., Darriet, P., Pons, A., 2025. Oxygen transfer rate measurements of closure systems after 12 years of aging. Impact on the aroma compounds of sauvignon blanc wines. Food Packag. Shelf Life 48, 101452. https://doi.org/10.1016/j.fpsl.2025.101452.

Tedjditi, A.K., Ghomari, F., Taleb, O., Belarbi, R., Tarik Bouhraoua, R., 2020. Potential of using virgin cork as aggregates in development of new lightweight concrete. Constr. Build. Mater. 265, 120–734. https://doi.org/10.1016/j.conbuildmat.2020.120734.

Ureña, M., Chanut, J., Bottreau, V., Bellat, J.-P., Gougeon, R.D., Lagorce, A., Karbowiak, T., 2024. Wine bottle overcapping wax: an aesthetic or functional element? Food Packag. Shelf Life 46, 101367. https://doi.org/10.1016/j.fpsl.2024.101367.

Xia, Y., Yang, X., Zhou, Q., 2009. Effect of stress softening in bumper foams on the low speed impact performance of vehicles. SAE Int. J. Mater. Manuf. 1 (1), 548–553. ⟨https://www.jstor.org/stable/26282688⟩.

Yang, X., Xia, Y., Zhou, Q., 2011. Influence of stress softening on energy-absorption capability of polymeric foams. Mater. Des. 32 (3), 1167–1176. https://doi.org/10.1016/j.matdes.2010.10.024.