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How to get reliable oxygen transmission rate values for coated cardboards?

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ABSTRACT

Coated papers have emerged as alternatives to petroleum-based and non-biodegradable plastics, particularly in packaging industries. Indeed, barriers brought by the coating layer, toward oxygen for example, allow better food preservation. This growing interest in the field of packaging has led to the need to characterize the mass transfer properties of these materials, particularly oxygen. Although methods and standards to characterize these parameters have been developed for plastic materials, none have been specifically adapted for coated cardboards. This paper evidences a so-called "edge effect" occurring when characterizing the oxygen transmission rate (OTR) of single-side coated cardboards. This leads to two significantly different OTR values depending on the material's side, cardboard, or polymer coating, exposed to the oxygen flux. Over- or under-estimated OTR can have a significant impact on food preservation. Therefore, avoiding edge effects is essential for obtaining accurate oxygen transmission rate value. This study focuses on one measurement method and two cardboard supports. Depending on the equipment used and sample properties, different methods can be used to prevent edge effects and must be investigated on a case-by-case basis.

1. Introduction

In the field of food packaging, cellulosic materials such as paper and cardboard appear to be excellent alternatives to conventional fossil-based plastics that persist in our environment, provided that their very low barrier properties and highly hydrophilic nature are overcome while maintaining their recyclability and biodegradability. Papers and cardboards are thus combined with thin layers of high-barrier and water-resistant biopolymers [1–5] coated on one or both sides.

The main role of packaging materials is to ensure food preservation and protection. Because each food category has different requirements, the optimal packaging specifications are different for each food product. In modified atmosphere packaging (MAP), the key property of packaging materials is mass transfer. Controlling this factor is essential for the reduction of food waste and losses. Therefore, it is necessary to characterize the barrier properties of these materials accurately.

One main barrier property is measured through the Oxygen Transmission Rate (OTR), which characterizes the quantity of oxygen (O_2) passing through a unit area of a film per unit time under test conditions. In the steady state, the oxygen transmission rate OTR (mol.m^{-2}.s^{-1}) is defined by the following equation (Eq. (1)):

$$OTR = \frac{J}{A} \quad (1)$$

where J (mol.s^{-1}) is the O_2 mass flow and A is the surface area of the film (m^2).

Several standards describe methods for this measurement that relies on an O_2 partial pressure gradient between both sides of the material [6]. Compared to plastic films, cellulose-based multilayered materials present specificities regarding their structure because they possess a highly porous and permeable cellulose layer, which can be thick in the case of cardboards. Moreover, if the coating is applied on only one side, the resulting coated papers and cardboards become asymmetric [7–10]. Thus, the question of the sample's orientation in relation to the O_2 partial pressure gradient when conducting OTR measurements can arise.

To the best of our knowledge, in the literature, only four articles indicated the sample's orientation: Vartiainen et al. (2004) and Hult et al. (2013) placed the coated side toward the permeant gas whereas

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Hirvikorpi et al. (2010) and Kääriäinen et al. (2011) oriented the coated side to the carrier gas, [10–13] but no one explained nor mention what could be the expected impact on the resulting OTR. No study on the impact of side exposure on the resulting OTR has been found in the available literature. Thus, there is a gap in the literature regarding recommendations for OTR measurements of asymmetric coated cardboards, notably regarding the sample orientation in the permeation cell.

In this context, the objective of this paper was to investigate the impact of the orientation of single-side coated cardboards during OTR measurement on the resulting OTR value and its reliability, and to propose recommendations. For this purpose, single- and two-sides structures were tested in both orientations. Cardboards coated with polyvinyl alcohol (PVOH) on one or two sides were compared to uncoated porous cardboard, a nonporous multilayer polyethylene/polyamide (PE/PA) film, and a nonporous monolayer low-density polyethylene (LDPE) film. An accumulation method using an O_2 sensor was used to measure the OTR and as a reference method, as it is closer to the reality of packaging materials: one side of the sample faces the accumulation chamber, with no flux, while the other side faces the O_2 flux. During food storage, the inside of the packaging is confronted with a closed static environment, whereas the outside is often confronted with an environmental flux.

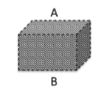
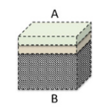
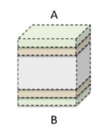
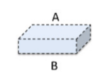
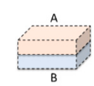
2. Materials & methods

2.1. Materials

The cellulose-based materials, i.e., uncoated cardboard (UC) and coated cardboards (CC), were provided by the Centre Technique du Papier (CTP, Grenoble, France). Coated carboards (CC) were based on bleached Cupforma Natura and unbleached CKB Nude grades from Stora Enso (Sweden), with basis weights of 260 and 230 g.m^{-2} and thicknesses of 370 and 445 μm, respectively. Both cardboards were made of a three-layer fiber construction with chemi-thermomechanical pulp in the middle layer. Concerning the uncoated cardboard (UC), only the Cupforma Natura grade was studied in this paper. Two types of coated cardboards were considered: a single-side coated cardboard (CC-1s), corresponding to the Cupforma Natura cardboard with a successive deposition, on the same side, of two layers of polyvinyl alcohol (PVOH), of 5 and 3 g.m^{-2} respectively, and a two-sides coated cardboard (CC-2s) based on CKB Nude cardboard with two PVOH layers of 5 g.m^{-2} and 3.4 g.m^{-2} on one side and two PVOH layers of 4.6 g.m^{-2} and 3.5 g.m^{-2} on the other side. For both samples, the PVOH layers were coated using an 11 % w/w PVOH solution prepared with Poval 15–99 (Kuraray, Japan). Both coated cardboards were then subjected to fatty acid chloride chromatogeny on both sides using a method patented by the Centre Technique du Papier to bring hydrophobicity [14].

A 50 μm thick low-density polyethylene (LDPE) monolayer film (BBA emballages, Lunel, France) and a polyethylene/polyamide (PE/PA) bilayer film, with thicknesses of 80 μm for PE and 15 μm for PA (Domenico de Lucia S.p.a., Italy), were used as benchmarks for dense nonporous structures. The codification and characteristics of the materials are listed in Table 1.

Table 1
Materials' characteristics.

Codification	Composition	Structure Sides A and B identified	Coating weight (g.m^{-2})	Total thickness (μm)
UC	Uncoated cardboard (Cupforma Natura)		n/a	348 ± 7
CC-1s	Uncoated cardboard (Cupforma Natura) + 2 layers of PVOH on the same side		Side A: 5 + 3 Side B: no coating	354 ± 3
CC-2s	Uncoated cardboard (CKB Nude) + 2 layers of PVOH on each side		Side A: 5 + 3.4 Side B: 4.6 + 3.5	433 ± 18
LDPE	Low density polyethylene		n/a	49.7 ± 0.9
PE/PA	Polyethylene/ Polyamide		n/a	98 ± 3

n/a: not applicable.
N.B.: Schemes do not represent the real thickness of each layer.

2.2. Methods

2.2.1. Oxygen Transmission Rate (OTR)

2.2.1.1. Sample preparation. Samples were conditioned at 0 % RH for 48 h prior to characterization. A maximum RH of 5 % was observed in the desiccator. 50 % RH and 0 % RH are the most often conditions of relative humidity found in the literature. 0 % RH was used to avoid any impact of cardboard swelling. The film thickness was measured at five points equally distributed on the sample using a hand-held micrometer with a resolution of 1 μm (Digimatic micrometer 0-25 mm, Mitutoyo Corporation, Kawasaki, Japan).

2.2.1.2. Measurement. Samples were mounted between two metallic chambers. The sample surface was reduced by a metallic adapter with an open circular area of 12.6 cm^2, as it is recommended for highly permeable materials in ASTM standards [15,16]. Tightness was ensured by an O-ring seal, both between the two chambers and the metallic adapter and between the metallic adapter and the sample (Fig. 1). The permeability cell was placed in a thermo-regulated oven (IPP 500, Memmert, Germany) at 23 °C and connected to a temperature probe (Pt100, PreSens GmbH, Germany) and a Polymer Optical Fiber for Use with Minisensors (PreSens GmbH, Germany). Both probes were connected to a Fibox Trace 4 (PreSens GmbH, Germany) to record the temperature and O_2 quantity in the upper chamber, which was determined using an integrated O_2 sensor (PSt6, PreSens GmbH, Germany). Prior to measurements, both the upper and bottom chambers were flushed with dry N_2 until 0 % O_2 was detected in the upper chamber. The upper chamber was then closed, and a continuous 100 % dry O_2 stream was injected through the bottom chamber (with a flux of approximately 30 ml.min^{-1}). The amount of O_2 passing through the sample and arriving in the upper chamber was measured every 3 s until a value of 2 % was reached. For high barrier materials, such as PE/PA, measurements were performed until 0.5 % O_2 was reached in the upper cell to decrease the measurement time. This method was therefore an accumulation method, with no flux on one side of the sample and a continuous O_2 flux on the other side.

2.2.1.3. OTR calculation. The O_2 permeability was determined by resolving Fick's first law, with the flux expressed in mol.s^{-1}, given by the following differential equation (Eq. (2)):

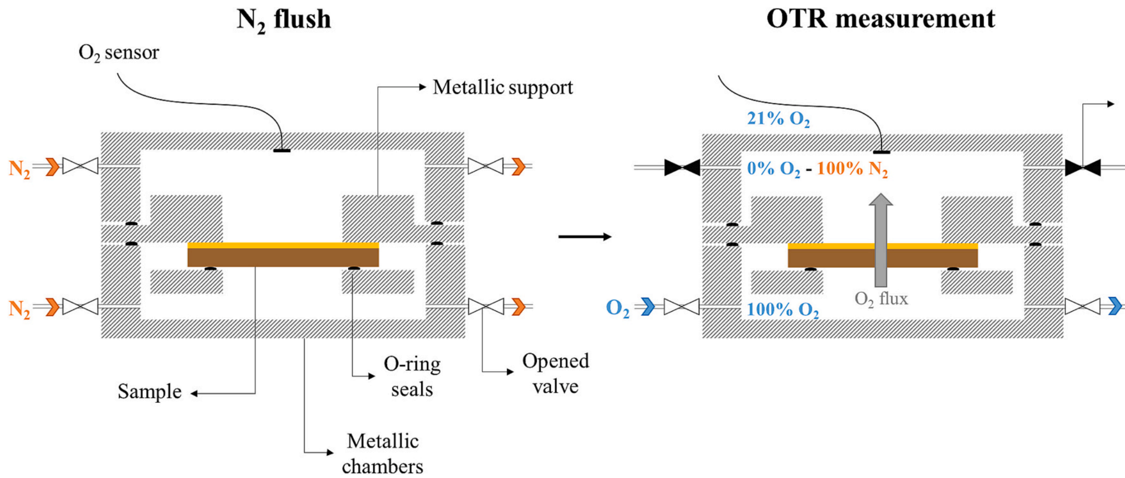


Fig. 1. Scheme of the OTR measurement equipment and principle.

$$\frac{dn}{dt} = P \times \frac{A}{l} \times (p_{low} - p_{up}) \quad (2)$$

Where n is the O_2 quantity in the upper cell (mol), P the material's permeability ($mol.m.m^{-2}.s^{-1}.Pa^{-1}$), A the sample's surface area (m^2), l the sample's thickness (m), p_{low} the constant O_2 partial pressure in the lower cell (Pa), and p_{up} the O_2 partial pressure in the upper cell (Pa), which is proportional to n according to the ideal gas law. The estimated parameter, P, was estimated by minimizing the Root Mean Square Error between the experimental and simulated O_2 quantities in the upper cell. The numerical solution was obtained using a MATLAB (The MathWorks Inc., Natick, Massachusetts, USA) code based on ode15s and lsqnonlin functions [17].

Considering an O_2 partial pressure gradient of 1 atm, the permeability values were then converted to OTR ($cm^3.m^{-2}.day^{-1}$).

2.2.2. Sample orientation in the permeability cell

The orientation of the material was defined according to the sides A and B (Table 1) of the material facing the sensor. Case A corresponded to side A facing the sensor; therefore, side B faced the permeating O_2 flux, whereas case B corresponded to the side B facing the sensor; therefore, side A faced the permeating O_2 flux. For UC and LDPE films that were delivered in reels, side A was the side toward the inside of the reel, whereas side B corresponded to the side toward the outside of the reel. Three replicates for each orientation were performed, except for LDPE and PA/PE, which were industrially produced and more repeatable, and therefore performed in duplicates for each orientation.

3. Results & discussion

3.1. Evidence of a side effect for single-side coated cardboards

Oxygen barrier properties (OTR) were characterized for uncoated (UC), single-side (CC-1s), and two-sides (CC-2s) coated cardboards, LDPE, and PE/PA films. As main result, there was a difference between the OTR values obtained for CC-1s depending on the case considered. Indeed, there is a ratio of OTR_{CaseB}/OTR_{CaseA} over 7 between the two values, evidencing what was called a "side effect" (Fig. 2). However, this side effect was not observed for the two-side-coated cardboard (CC-2s) nor for the dense polymeric films (LDPE and PA/PE), where the ratio values were close to 1. For the uncoated cardboard (UC), the OTR values for cases A and B did not show a side effect, but no strict conclusion can be drawn since both values were out of range, owing to the high permeability of the cardboard (> 5.10^6 $cm^3.m^{-2}.day^{-1}$). The comparison of the materials allowed to draw some preliminary conclusions. As dense mono-materials (LDPE) showed no side effects, this phenomenon

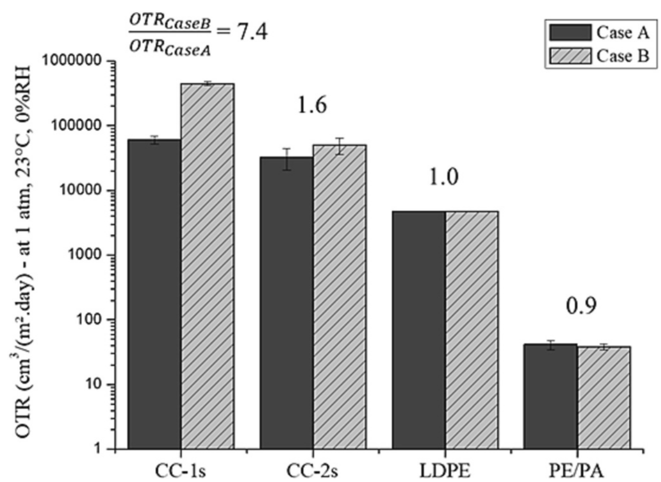


Fig. 2. OTR values of the materials for both case A and case B (doi: 10.57745/YR3HRI).

was associated with multi-material structures and/or the presence of porous materials. However, among the characterized multi-materials, dense multi-material structures, such as PE/PA, did not show side effects, highlighting the importance of the presence of a porous layer in the multi-layer structure. CC-2s showed the same value in both cases, indicating the importance of asymmetry. Thus, it was concluded that the side effect was associated with asymmetric multilayer materials, containing at least one porous layer. Measurements performed on other asymmetric coated cardboards at 50 % RH confirmed this side effect on the asymmetric multilayer materials, containing at least one porous layer (Table S2). Regarding porous monolayer materials, no side effect is expected owing to the similarity between the two sides of the material. However, the out-of-range values measured in this study did not allow to conclude on this.

Considering OTR_{CaseB} or OTR_{CaseA} can lead to two different decisions when choosing packaging for a specific product. In a modified atmosphere system, where gas fluxes are paramount for food protection, an error of a factor of 7 on the OTR will lead to an error of the same factor on the flux thus hampering the shelf-life of the product.

To our knowledge, no article in the literature has studied the orientation of the material in OTR measurements; thus, none of them evidence a side effect, and the ratio observed between case A and case B cannot be compared to literature values.

An explanation for this observation was searched for in ASTM standards dedicated to oxygen or gas permeability measurements (Table 2), which include cardboards and papers in their scope. Indeed, cardboards and papers, coated or not, are often mentioned in the scope of the ASTM standards, demonstrating their relevance for such materials. In the literature, when characterizing OTR of multilayered asymmetric coated cardboards, most authors followed the ASTM D 3985 standard [8–10,18–21]. Others mentioned the DIN 53380-3 standard [14], or used their own methods [7,13,22]. Most ASTM standards make recommendations for highly permeable and asymmetric materials, which include coated cardboards. For the class of highly permeable materials (i.e., materials with transmission rates >200 cm^3(STP).m^{-2}.day^{-1}), e.g. some cellulose-based materials, it is often suggested to reduce the O_2 flux over time to characterize it more accurately. This can be achieved by decreasing the concentration of O_2 in the permeating gas flow [15,16,23,24] and/or decreasing the surface area of the sample [15,16]. In this paper, the second option was selected, as an adapter was used to reduce the surface area. For asymmetric materials, some standards specify that two sides can be identified and, more importantly, mention the need to identify these two sides and report the orientation of the specimen in the diffusion cell (i.e., the side exposed to the permeant flux) [15,16,24–26].

However, as for articles in the literature, no explanation was found in those standards regarding the impact of such orientation on the resulting OTR values or whether an exposed side, either coated or not, is more adequate. Thus, no indication is given on the correct value between Case A and Case B, nor explain the reason for obtaining different values between both orientations.

As the OTR value directly impacts the field of application of materials, this side effect must be further investigated and understood.

3.2. Elucidating the side effect

Three hypotheses were proposed to explain the side effect observed for single-side-coated cardboards:

Hypothesis 1. The residual quantity of O_2 dissolved in the cellulosic substrate, prior to measurements (due to contact equilibrium with the surrounding atmosphere), is suddenly released at the beginning of the OTR measurements, leading to an overestimation of the initial O_2 flow and thus, an overestimation of the OTR. As CC-1s is asymmetrical, the oxygen desorption kinetics should be different from case A to case B. Indeed, for case B, the dissolved oxygen can be released directly into the measurement chamber, faster than in case A where it must pass through the PVOH layer to be detected by the sensor.

Hypothesis 2. An additional O_2 transfer occurs through the edges of the samples when the uncoated side is exposed to the sensor, i.e., case B, which increases the overall O_2 flux, leading to an overestimation of the OTR under that condition. O_2 can pass through the edges of the sample, despite the O-ring seals, owing to the porosity of the cardboard. Indeed, edges of the sample are in contact with 100 % O_2, due to the cell configuration (Fig. 1). When the PVOH coating faces the sensor (case A), O_2 passing through the edges must still pass through the PVOH barrier layer. However, when the cardboard faces the sensor (case B), O_2 passing through the edges can reach the upper chamber only via the porous cellulosic layer. In the latter case, this would result in more O_2 being detected in the upper chamber (Fig. 3). Some industries recommend using specific cells or configurations to avoid edge effects on coated cardboards.

Hypothesis 3. The OTR of asymmetric coated cardboards is intrinsically different depending on which side of the material is exposed to O_2 flux. Considering an equivalent homogenous material following Fick's

Table 2
Summary of ASTM (American Society for Testing and Material) standards related to the characterization of oxygen permeability of film sheets or packaging.

Name	Standard	Permeability toward	Method	Comments on high permeable materials	Comments on asymmetric materials
Standard Test Method for Oxygen Gas Transmission Rate through Plastic Film and Sheeting using a Coulometric Sensor	D3985-17	O_2	Coulometric	• Use a gas mixture with a lower migrant concentration Or • Use a mask to reduce the surface area	If asymmetrical construction: • Identify and distinguish the materials faces. • Report the material's orientation in the diffusion cell
Standard Test Method for Determination of Oxygen Gas Transmission Rate, Permeability and Permeance at Controlled Relative Humidity Through Barrier Materials Using a Coulometric Detector	F1927-14	O_2	Coulometric	Use a gas mixture with a lower migrant concentration	
Standard Test Method for Oxygen Gas Transmission Rate through Plastic Film and Sheeting using Dynamic Accumulation Method	F3136-15	O_2	Accumulation	/	
Standard Test Method for Oxygen Gas Transmission Rate through Plastic Film and Sheeting using Various Sensors	F2622-20	O_2	3 configurations: - Background Gas Cylinder OTR - Background Diffusion Cell OTR - Measured Diffusion Cell OTR	• Use a mask to reduce the surface area • Use a gas mixture with a lower migrant concentration	
Standard Test Method for Oxygen Transmission Rate Through Packages Using a Coulometric Sensor	F1307-20	O_2	Coulometric		/
Standard Test Method for Film Perme ability Determination Using Static Permeability Cells	E2945-14 (Reapproved 2021)	All gases/ fumigants (gas, solid, liquid)	Static	Increase measurements frequency	If asymmetrical construction, report the material's orientation in the diffusion cell
Standard Test Method for Determining Gas Permeability Characteristics of Plastic Film and Sheeting	D1434-82 (Reapproved 2015)	All gases	Manometric and Volumetric	/	/

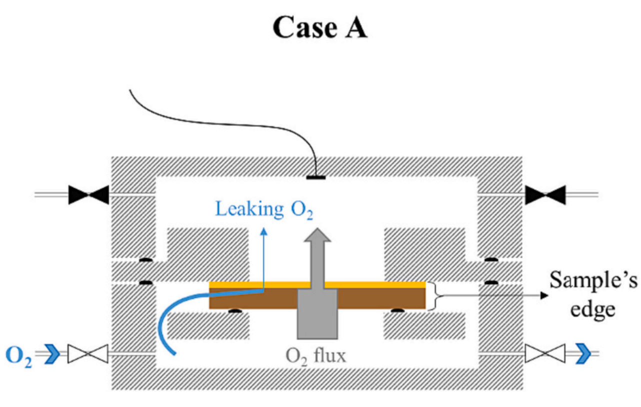


Fig. 3. Edge effect hypothesis scheme for case A (left) and case B (right).

law, the apparent permeability is the product of apparent diffusivity and solubility and therefore should be identical regardless of the side facing the O_2 flux. Therefore, the O_2 transfer of these asymmetric and porous materials would not be Fickian, or these materials would have different O_2 diffusivities or solubilities, depending on the orientation.

3.2.1. Hypothesis 1

Theoretical calculations were used to determine the amount of oxygen dissolved in the coated paper. Based on basis weight and thickness values, UC's apparent porosity was calculated according to the following equation (Eq. (3)):

$$\varphi = 1 - \frac{bw}{l \times d_{cellulose}} \tag{3}$$

where φ is the cardboard apparent porosity, l is the thickness of the cardboard (cm), bw is the cardboard's basis weight (g.cm^{-2}) and $d_{cellulose}$ is the cellulose density (considered equals to 1.5 g.cm^{-3}).

The calculated porosity of Cupforma cardboard was 0.5. The worst case was considered, where all pores were initially filled with pure O_2, and this whole quantity, 9.6×10^{-6} mol of dissolved O_2, was totally released in the measurement cell, neglecting the O_2 desorption during the N_2 flush. Therefore, a corrected OTR value was calculated when the paper was exposed to the sensor (case B) by removing the dissolved O_2 flux from the total O_2 flux passing through the sample (Fig. 4). For case B, the corrected OTR value was 18 % lower than the one obtained experimentally but remained higher than the OTR value measured for case A, with a ratio between OTR_{CaseB} and OTR_{CaseA} equals to 6.1. Thus, it was concluded that this correction did not explain the side effect.

3.2.2. Hypothesis 2 & 3

To test the second hypothesis, assuming that there are edge effects responsible for overestimations of OTR results when the cardboard side faces the sensor (Fig. 3), CC-1s samples were sandwiched between two masks made of aluminum adhesive, maintaining a surface area of 12.6 cm^2. By covering the edges of the asymmetric samples with adhesive aluminum masks, no O_2 could pass through the edges. The sandwiched samples were then mounted in the metallic adapter, and measurements were performed under the same conditions as the unmasked samples. Three replicates were done for both case A and case B. As shown in Fig. 5, when the sample edges were covered with an adhesive aluminum mask, OTR values of the CC-1s samples were not significantly different for cases A and B. Therefore, these observations confirmed that edge effects were mostly present when the porous cardboard side was facing the sensor, O_2 reaching the upper chamber without passing by the PVOH layer and led to overestimation of the O_2 flow and thus, OTR. Such aluminum masks have already been used in the literature [7–9,17,20,21], but none of them mention edge effects as a reason to use such masks.

It is also worth noting that such edge effects must occur during OTR measurements of uncoated cardboards, as they represent porous materials; however, it was not possible to evidence them in this case because the uncoated cardboard is too permeable.

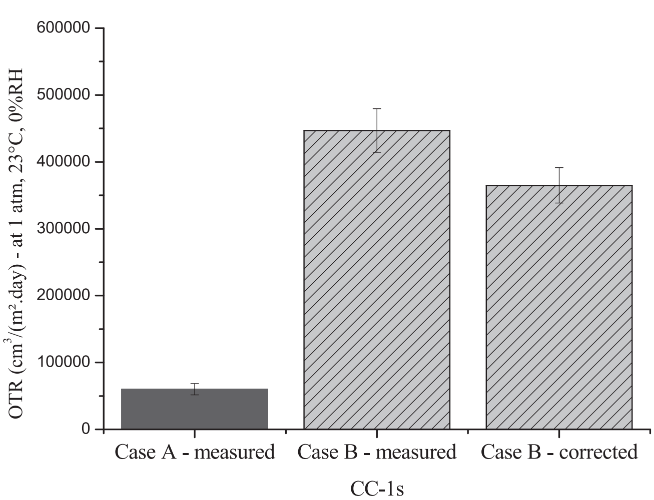


Fig. 4. Comparison between experimental and corrected (subtracting the theoretical dissolved O_2 quantity to the overall permeation flux) values of CC-1s OTR for case A and B.

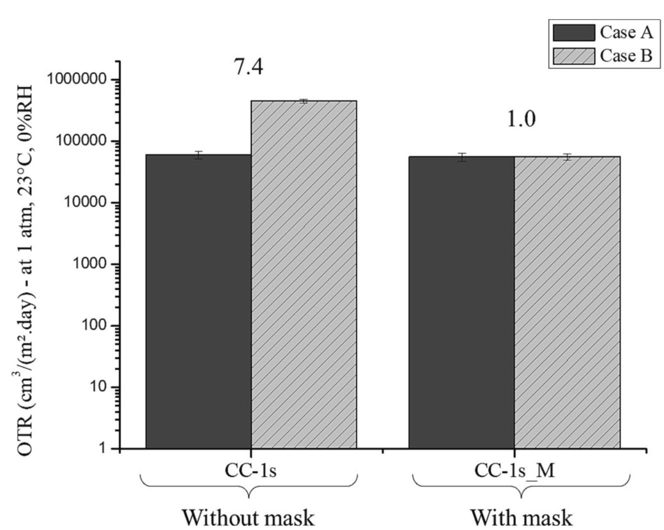


Fig. 5. OTR results for CC-1s material using adhesive aluminum masks.

At the same time, validating the second hypothesis automatically rejected the third hypothesis since the same value was found for cases A and B when adhesive aluminum masks were used. This confirms that asymmetric coated cardboards do not have two OTR values depending on the orientation of the material toward the flux.

4. Conclusion

In this study, OTR measurements of five different materials highlighted a side effect when the material was porous and asymmetric, resulting in two different OTR values depending on the material's side exposed to the O_2 flux (seven times higher for case B than A). This side effect did not occur for symmetric porous and dense materials nor for asymmetric dense materials.

Various hypotheses regarding this effect have been investigated. The O_2 trapped in the cardboard layer did not explain the observed difference between the OTR values of case A and case B. It has been inferred that the difference was due to the leaking gas penetrating through the cardboard layer, the so-called edge effect. This edge effect did not occur when an aluminum mask covering the edges of the sample was used. This also confirmed that the initial difference observed in the OTR was not an intrinsic property of the asymmetric cellulosic material, but an experimental artifact. Thus, this paper demonstrates the importance of removing edge effects when characterizing the mass transfer properties of asymmetric porous materials. Depending on the equipment used and sample properties, the edge effects can be removed, for example by covering the edges with an aluminum mask, epoxy resin, or a specific film test cartridge for instance. Before each testing campaign, tests must be performed to ensure the efficiency of the method.

Edge effects were evidenced on coated cardboards with two different thicknesses and porosities. It would be interesting in future studies to carry out a deeper investigation to determine the impact of cardboard thickness and porosity on these effects. Even if this deeper study would be helpful, the interdependence between cardboard porosity and thickness makes it difficult to obtain cardboards with the same thickness but with different porosities, and vice versa. Indeed, to conduct such studies on the impact of cardboard properties (i.e., porosity and thickness) on the edge effect, the obtention of such specific cardboards is mandatory.

CRediT authorship contribution statement

Emma PIGNERES*: Conceptualization, Data curation, Data analysis, Investigation, Methodology, Roles/Writing – original draft

Allison VERCASSON*: Conceptualization, Data curation, Data analysis, Investigation, Methodology, Roles/Writing – original draft

*Both co-first authors, PIGNERES and VERCASSON, have contributed equally to this work.

Sébastien GAUCEL: Conceptualization, Funding acquisition, Methodology, Supervision, Validation, Writing – review & editing

Fanny COFFIGNIEZ: Conceptualization, Funding acquisition, Methodology, Supervision, Validation, Writing – review & editing

Nathalie GONTARD: Conceptualization, Funding acquisition

Hélène ANGELLIER-COUSSY: Conceptualization, Funding acquisition, Methodology, Supervision, Validation, Writing – review & editing

Valérie GUILLARD: Conceptualization, Funding acquisition, Methodology, Supervision, Validation, Writing – review & editing

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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.

Data availability

How to get reliable oxygen transmission rate values for coated cardboards? (Original data) (Dataverse)

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.porgcoat.2023.108048.

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