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# Assessing the suitability of new film laminates for sustainable insect eradication by modified atmosphere in museums

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## Abstract

The increasing demand for applying modified inert atmosphere systems for insect eradication in museums has led to the desire for lower-cost consumable materials, particularly laminated plastic films. An ultra-low oxygen-permeable laminate is required for creating successful systems of modified inert atmosphere to keep the oxygen concentration lower than 0.3%. A range of ultra-high barrier laminates is commercially available but at a high cost, which could affect the sustainability of applying these systems in museums of limited budgets. The wide use of local laminates for food preservation makes them a perfect target for testing and improvement as alternatives. However, a laboratory test method for evaluating their oxygen permeability is required to assess their suitability for insect eradication using modified atmospheres. Therefore, the present work investigates the potential of using two laminates (one local and one imported) to create a successful modified atmosphere system. A laboratory easy-to-use test method was used to assess the oxygen permeability of each laminate and calculate its oxygen transmission rate. The test method is a sealed static chamber separated in the middle by a known area of the laminate to be tested. The test relies on monitoring the change of oxygen concentration overtime on either side of the laminate membrane within the sealed system to assess its oxygen transmission rate. The specifications and design of the test chamber are adopted from the ASTM Designation: E2945 – 14. Results indicated that the new method is useful for an unlimited number of tests of an unlimited number of laminates. The conducted tests proved that the local laminate normally used for food packaging showed good results in terms of its oxygen transmission rate.

**Keywords:** Plastic film, Oxygen transmission rate, Insect disinfection, Museums pests, Permeability test, ESCAL, Nylon, Cross-barrier, Low-oxygen

## Introduction

The preservation of cultural and historical collections from insect attack is a challenging task, as insects still are a primary cause of loss of heritage materials [1]. Modified inert atmospheres (MIAs) where oxygen is intentionally reduced by an inert gas [2] are widely used for eradicating insect pests [3–6], significantly slow decay processes [7], and allow for long-term preservation of heritage objects

[8, 9] since the 1990s [1]. The treatment can be applied in rigid chambers, portable bubbles, or low-permeability plastic bags [10]. The use of flexible plastic bags that fit the shape and size of the infested collection is generally a practical solution [11]. The plastic films used to create MIA bags must meet some criteria. A typical film must be flexible to easily create custom-made bags or tents, heat sealable to one another at a surface temperature not affecting the surface of the other laminate, non-puncturable or non-scratchable, transparent enough to see the artifacts through them, low permeable to oxygen, and available at low cost to ensure sustainable application of the treatment.

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The properties of single-layer plastic films are affected by many factors, for example the chemical nature of the film polymer, the distribution and concentration of its molecules, and the fabrication process itself [12]. Although additives are used for optimizing some properties [13–15] or adding new ones to plastic films [15–19], no physical or chemical modifications resulted in a perfect, single-layer film that meets all the requirements for MIA applications [12]. Therefore, laminated films of special properties are used for MIA purposes. The MIA laminate is normally composed of three layers of polymer films [15, 20]: an internal heat-sealable layer [21, 22], an oxygen barrier layer [11], and a protective outer layer. However, there is not yet a perfect laminate that fulfills all the required criteria for MIA applications [12], and the selection of the suitable laminate depends generally on its oxygen transmission rate (OTR) as the key factor for MIA effectiveness. OTR is often expressed as the amount of oxygen gas (cm<sup>3</sup>) that can pass through a square meter of the film membrane in one day [23–25]. Nowadays, a wide variety of laminates are industrially available and commonly used for MIA treatments worldwide for their low OTR (Fig. 1). Most of these laminates are very expensive and are produced by companies located mainly in the United States, Japan, and Germany, and must therefore be imported, which might restrict their use and double their costs.

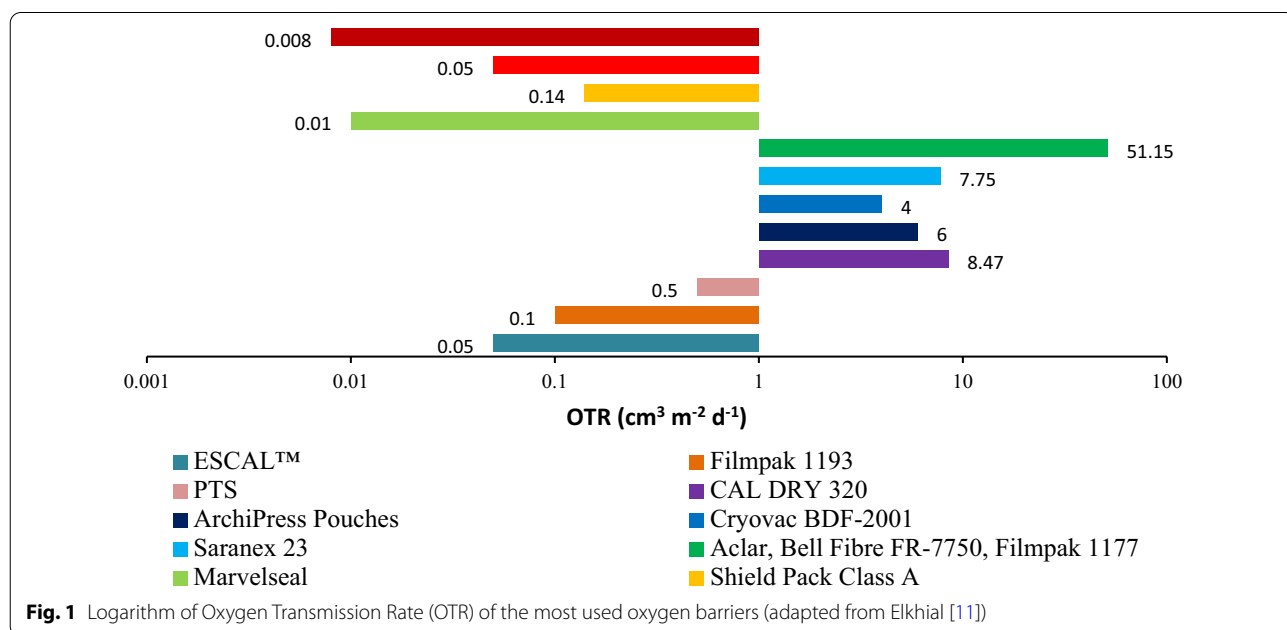
Depending on expensive laminates might therefore question the sustainability of applying MIA treatments in flexible bags in museums of limited budgets, and there is a need to look for local alternatives to decrease

the cost and guarantee sustainability. Fortunately, the food packaging industry yields a huge number of different local laminates. The only problem associated with their use is that their OTR might generally be unknown and they, therefore, need to be tested to assess their suitability for MIA applications. Automated permeability test devices that can be compatible with ultra-low permeable laminates might not be locally available. In 2001, a quick, reliable test method for measuring the transferred mass of the fumigants through plastic films was presented by Papiernik, et al. [26]. The method has been proved reliable and valid in testing low-permeable polyethylene films [27, 28] and was later standardized and approved by ASTM in 2014 [29] and updated in 2021 [30]. Accordingly, this study adopted and developed the standard test to provide an accurate method that could be easily applied in a museum laboratory to choose between the MIA laminates. The methodological approach has been used to assess the suitability of different laminates through measuring their OTR attempting to find alternatives to the expensive laminates to ensure the sustainability of applying MIA treatments in museums.

### Materials and methods

#### Plastic films

Three film laminates were selected in this study for measuring their oxygen permeability and OTR: a well-known MIA laminate, ESCAL™, and a local laminate in Japan, Cross-Barrier™, and a local laminate in Egypt, Nylon.



**ESCAL™**

ESCAL™ is manufactured by Mitsubishi Gas Chemicals Inc., Japan, and is distributed worldwide. It is produced in sheet and tube rolls and sachets. ESCAL™ is ceramic-coated polyvinyl alcohol (PVAL). The basic structure for this barrier film is orientated polypropylene (OPP) as a surface layer, silica deposited polyvinyl alcohol (SiO)<sub>x</sub> / PVAL as an intermediate layer, and linear low-density polyethylene (LLDPE) as an inner layer. It has superior oxygen and vapor barrier properties and its OTR is less than 0.1 cm<sup>3</sup> m<sup>-2</sup> d<sup>-1</sup> atm<sup>-1</sup> at 25 °C and 60% RH [31]. It is optically very yellow and structurally thick (114 μm) (Fig. 2a).

**Cross-barrier™**

The laminate is manufactured by Nissin Kasei Co., Ltd., Saitama, Japan. It is produced in sheet rolls. Its thickness is 145 μm and its weight is 115 g m<sup>-2</sup>. It was selected because it is a typical laminate consisting of three layers: a surface layer, an intermediate layer, and a back-side layer. The surface layer consists of aluminum oxide (AlO)<sub>x</sub> deposited on polyethylene terephthalate (PET). The intermediate reinforcement layer is typically made

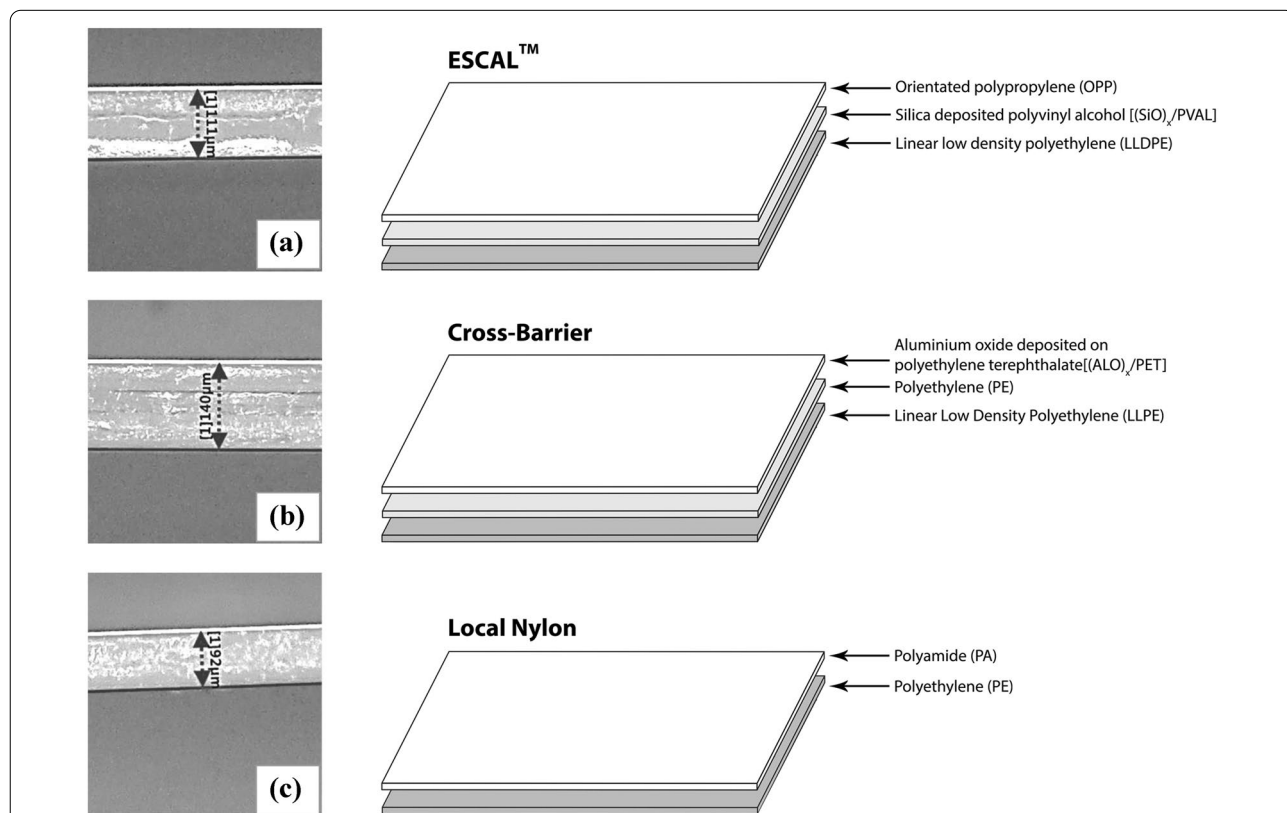
of polyethylene (PE). The back-side layer consists of linear low-density polyethylene (LLDPE). The composition ratio is approx. 13% PET, approx. 87% PE, and approx. 0.05% (AlO)<sub>x</sub> (Fig. 2b).

**Local commercial nylon film**

The laminate is manufactured by M2Pack Co., Cairo, Egypt. It is produced in different size sachets. The sachet used in this experiment measures 25 × 30 cm. Its thickness is 92 μm and its weight is approx. 77.33 g m<sup>-2</sup>. It was selected because it is a low-cost, widely locally available laminate consisting of two layers, a surface layer, and an inner layer. The surface layer consists of polyamide (PA). The inner layer is typically made of linear low-density polyethylene (LLDPE). The interesting fact about this laminate is that it is recyclable (Fig. 2c).

**Test chamber**

The test system consisted of two well-sealed chambers: a source chamber and a collecting chamber. The chambers were long and cuboid to suit all grades of permeable films (high to low) [27, 28]. The adopted design from ASTM [30] was modified by constructing the chambers



**Fig. 2** Structure of laminates. On the left, cross-sections of laminates at optical microscope at magnification ×200, with thicknesses in μm. on the right, schemes of stratigraphic structures

from 6 mm-thick plexiglass (Spiroplastic S.A., Cairo, Egypt) sheets for the availability and workability of the material in conservation laboratories in museums. The chamber walls were glued using chloroform. Each chamber had an internal volume of 3 L (10 × 10 × 30 cm), allowing a surface area of 100 cm<sup>2</sup> to attach the laminate membrane. One end of each chamber was closed by gluing it to a 10 × 10 cm piece of plexiglass. Holes with an 11-mm diameter were drilled in the test chambers at the mid-point height as shown in Fig. 3 to create the inlet and sampling ports. The holes were fitted with one ¼ in brass male O-seal connector (Swagelok® Company, Ohio, United States) and sealed off to the outer surface of the acrylic sheet with a washer. The inlet port was connected to a ¼ in two-way brass ball valve (Swagelok® Company, Ohio, United States) to allow or shut off the gas purge process and enhance sealing after purge (Fig. 4). At the sampling ports, two septa (Ametek Mocon, Inc., Minnesota, United States) were fixed and plugged with a ¼ in brass plug (Swagelok® Company, Ohio, United States) for sealing off the port (Fig. 5).

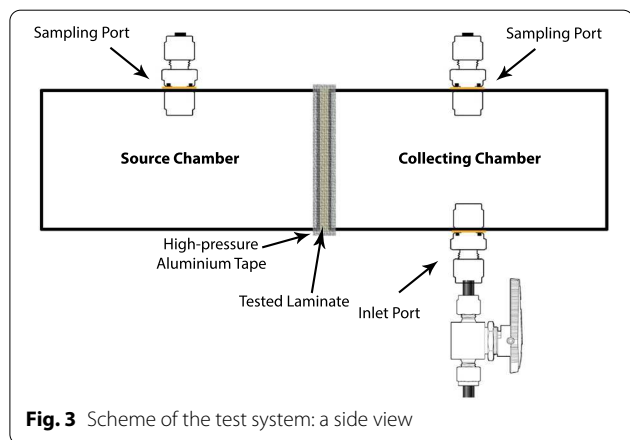
**Selection and source of inert gas**

Nitrogen was selected as the inert fumigant in this experiment because it is the most utilized MIA gas and the most difficult to maintain at the same time. The gas was obtained from a high-pressure prepurified T-size cylinder equipped with a Mujelli OX – AC one-stage regulator (GCE Group, Malmö, Sweden) with a built-in flow meter [11].

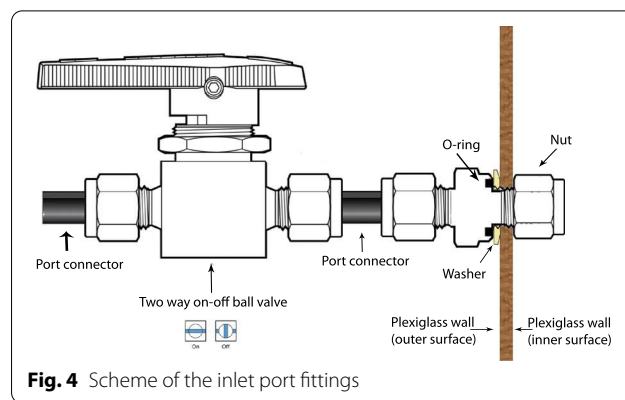
**Experimental procedure**

**Installation of laminates**

The tested film was glued to the open side of one chamber using epoxy glue mixed with its hardener and spread as a thin layer onto the 6-mm rim of the open side. After drying, a small amount of epoxy glue was evenly spread



**Fig. 3** Scheme of the test system: a side view



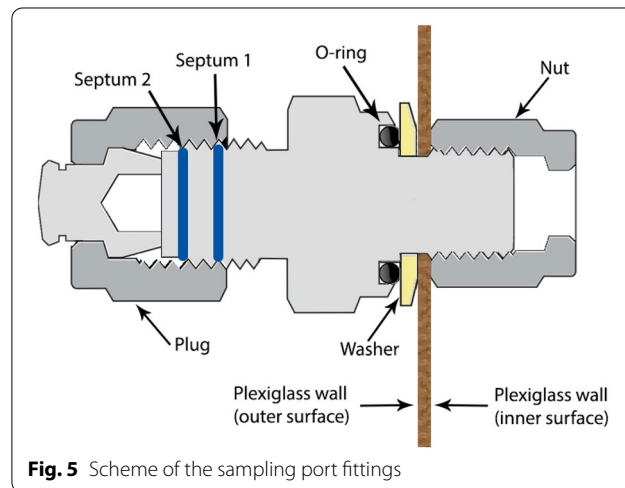
**Fig. 4** Scheme of the inlet port fittings

onto the rim of the other chamber that was directly placed onto the test film aligning and joining the two chambers together with the test film in the middle. The contact line between the two chambers was later tapped with a 2-cm wide strip of high-pressure aluminum tape to guarantee tight sealing.

After installation, the film laminates were conditioned for a minimum of 40 h at standard laboratory conditions (23 ± 2 °C and 50 ± 5% RH) immediately before testing according to ASTM D618-05 [32]. The conditions were controlled and monitored using the heating ventilation and air conditioning (HVAC) system of the fumigation laboratory, where the tests were performed.

**Gas purge sampling and analysis**

The source chamber was purged with pure, dry N<sub>2</sub> gas, following the dynamic purge process described by Elkhial [11], through the inlet port. After reaching 100% N<sub>2</sub>, the septa and plug were installed at the sampling port, followed by a quick shutdown of the ball valve then the gas



**Fig. 5** Scheme of the sampling port fittings

regulator to avoid pressure build-up and guarantee an O<sub>2</sub>-free gas path.

The concentration of N<sub>2</sub> was measured by monitoring the concentration of O<sub>2</sub> inside the bag using Dansensor® Checkmate® 3 Headspace O<sub>2</sub> analyzer (Ametek Mocon, Inc., United States), measuring range 0–100%, accuracy ±0.01%, and measuring three numbers after the decimal point. The utilized O<sub>2</sub> sensor uses the latest zirconia O<sub>2</sub> sensor, which has the inherent benefits of low cost, fast response, high sensitivity [33], and feedback control [34, 35]. The analyzer was calibrated using fresh air at 20.946% O<sub>2</sub>.

### Calculations and data processing

The oxygen permeability can be measured by a series of mathematical equations as given in many publications [27, 28, 30]. However, the standard provided a Windows-based software program, FilmPC (U.S. Department of Agriculture, California, United States), to facilitate the calculations by automatically solving the associated mathematical equations using a nonlinear least squares algorithm described by Marquardt [36] to obtain the parameters and statistics. To obtain the results in this study, FilmPC software, version 3.0.4, 2011 [37], was provided with the sampling time and concentration for each chamber sampled at the same time of the day. The initial concentration of oxygen in the source chamber was 0%. The initial value of oxygen in the collecting chamber was 20.9%, normalized to 100% in the graphs, and the loss of oxygen over time due to the transmission was recorded as the collecting chamber concentrations. After obtaining the results from the software, the OTR was calculated as it represents the permeability of oxygen over

time through 1 m<sup>2</sup> area of the film at specified conditions of temperature and relative humidity following the equation:

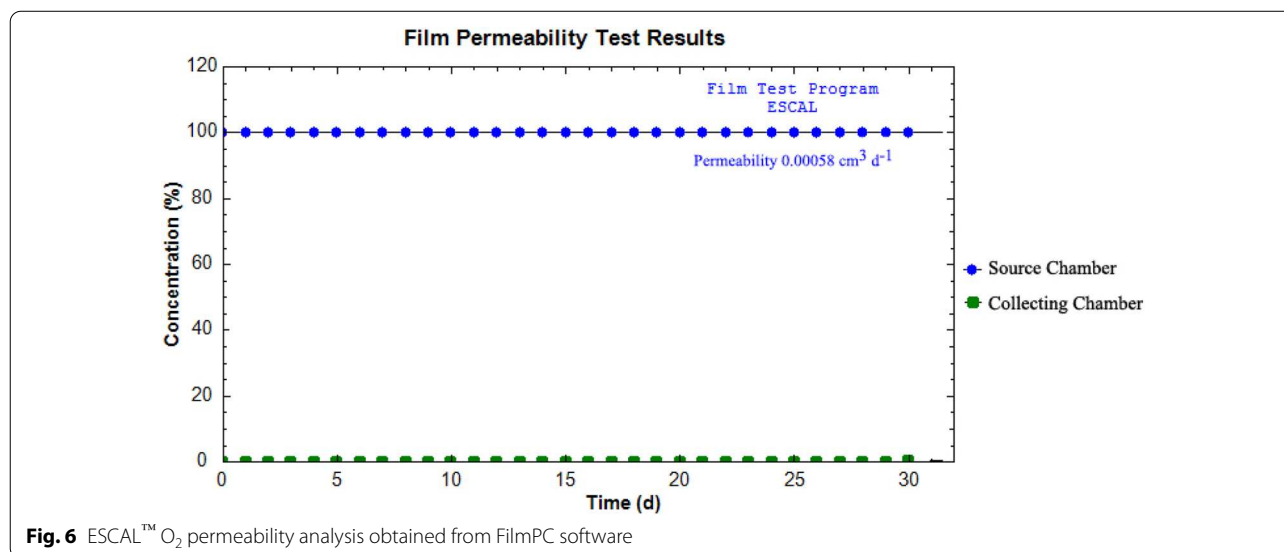
$$OTR = \frac{Q}{At}$$

where  $Q$  is the amount of permeant passing through the polymer (cm<sup>3</sup>),  $A$  is the area (m<sup>2</sup>) and  $t$  is the time (d) [38].

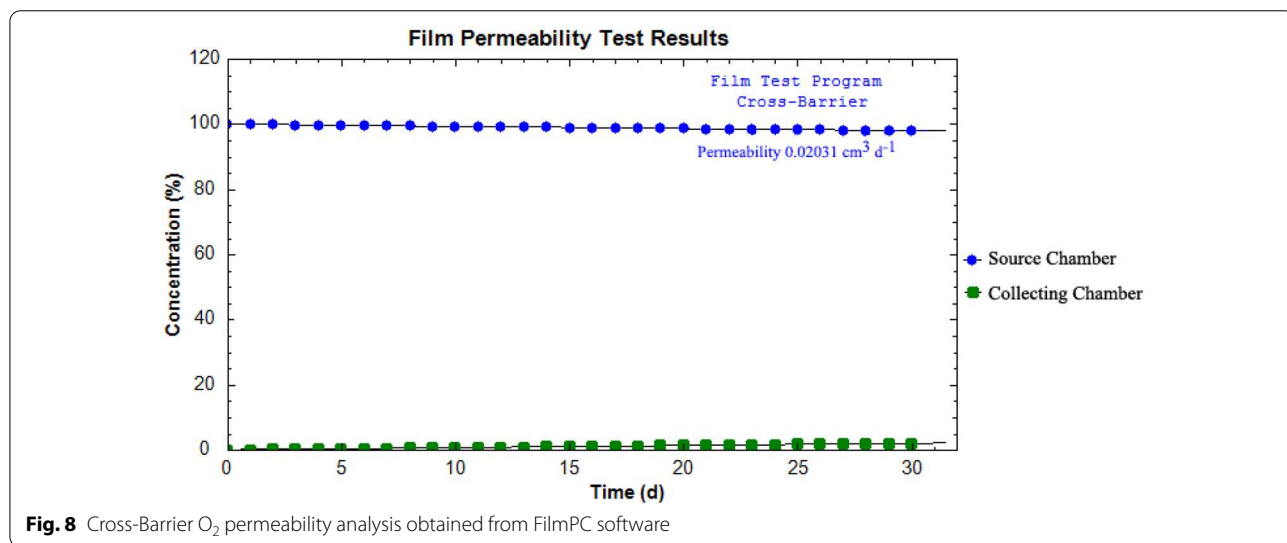
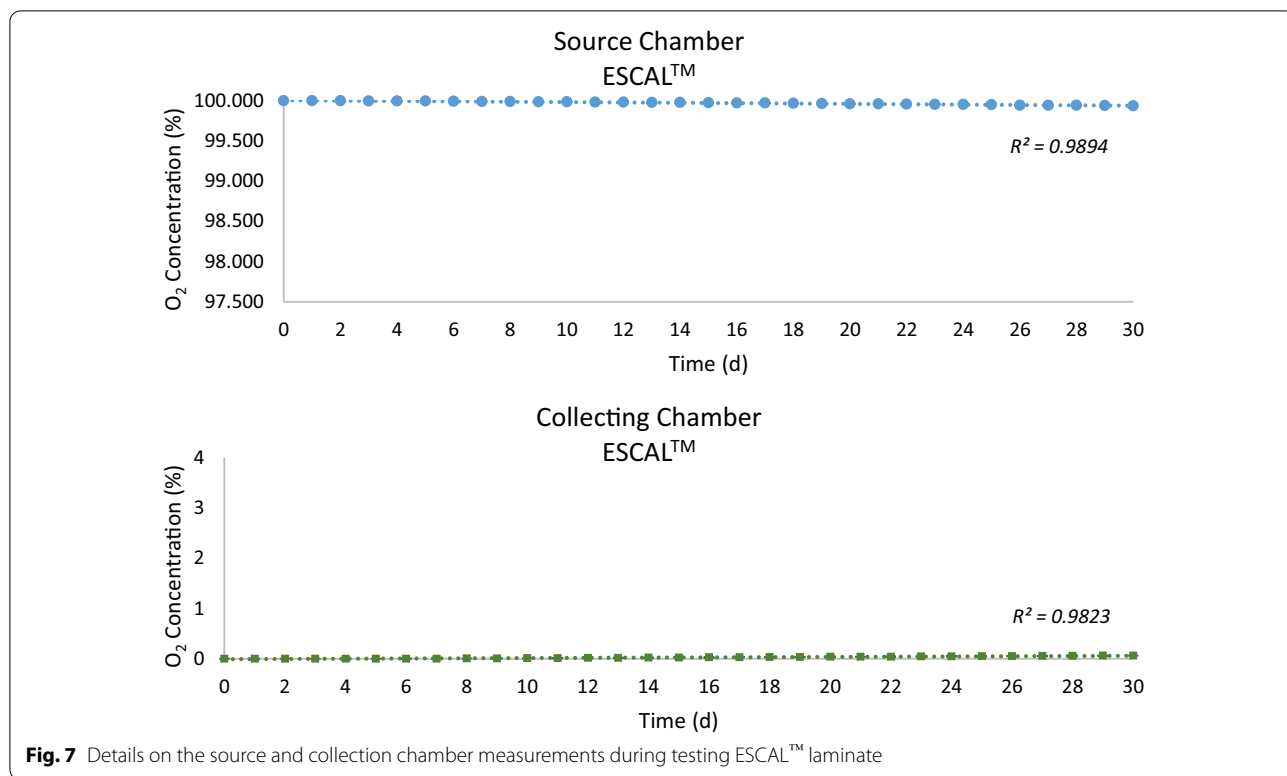
### Results and discussion

The oxygen permeability of three laminates was measured using a modified design of the test chamber approved by ASTM [29, 30] resulted in a completely airtight system and allowed for measuring very low oxygen permeability values. The oxygen permeability of the tested area of ESCAL™ laminate was  $0.00058 \pm 0.0001$  cm d<sup>-1</sup> (Figs. 6 and 7). The calculated OTR for a square meter is  $0.06$  cm<sup>3</sup> m<sup>-2</sup> d<sup>-1</sup>. Cross-Barrier data showed an oxygen permeability of the tested area of  $0.02031 \pm 0.0002$  cm d<sup>-1</sup> (Figs. 8 and 9) with calculated OTR for a square meter of  $2$  cm<sup>3</sup> m<sup>-2</sup> d<sup>-1</sup>. Nylon, however, showed oxygen permeability of  $0.04094 \pm 0.0005$  cm d<sup>-1</sup> (Figs. 10 and 11), with a calculated OTR of  $4.1$  cm<sup>3</sup> m<sup>-2</sup> d<sup>-1</sup>. The oxygen permeability graph of double-layered Nylon showed a rate of  $0.01925 \pm 0.003$  cm d<sup>-1</sup> of oxygen permeability which corresponds to  $1.9$  cm<sup>3</sup> m<sup>-2</sup> d<sup>-1</sup> OTR (Figs. 12 and 13).

Comparing the resultant OTR values of the tested laminates, the present study demonstrated the ultra-high oxygen barrier property of ESCAL™. The measured OTR of Nylon film showed that it is of exceptional quality, as generally the OTR of polyamides ranges from 18 to 40 cm<sup>3</sup> m<sup>-2</sup> d<sup>-1</sup> ([40] and goes up to 60 [39, 41] or 110



**Fig. 6** ESCAL™ O<sub>2</sub> permeability analysis obtained from FilmPC software

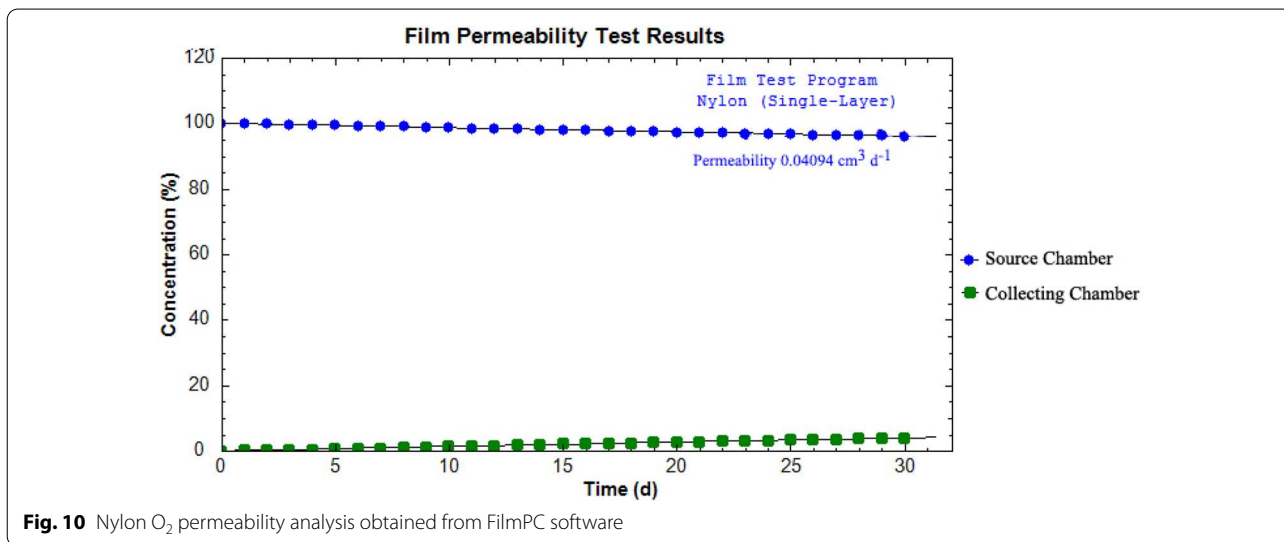
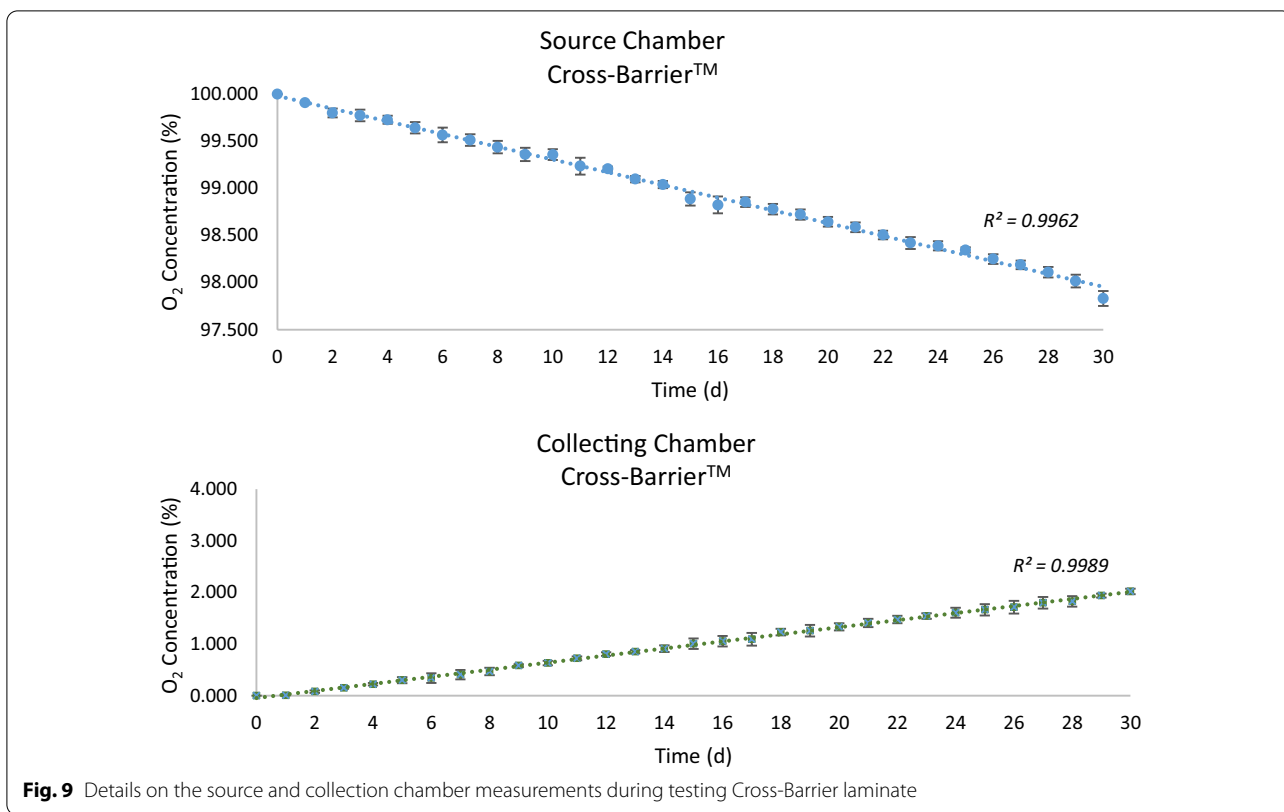


cm<sup>3</sup> m<sup>-2</sup> d<sup>-1</sup> [12, 42]. A superior result, however, was achieved by doubling the layer of Nylon, proposing it as a promising alternative to the expensive laminates. The cost of the doubled laminate was still far less than the cost of a single layer of the imported laminates (Table 1).

The methodological approach for testing the OTR of the film laminates is fairly straightforward and comes up with good results. The chambers are easy to create

and assemble and could be easily replicated using any inert, leak-proof materials. Additionally, the analyzer is easy to use, normally available in museums that apply MIA treatments, and available in modern versions to measure oxygen and carbon dioxide at the same time, allowing for performing two tests at once. The only apparent concern about using this system could be the possibility of having leakage areas in the

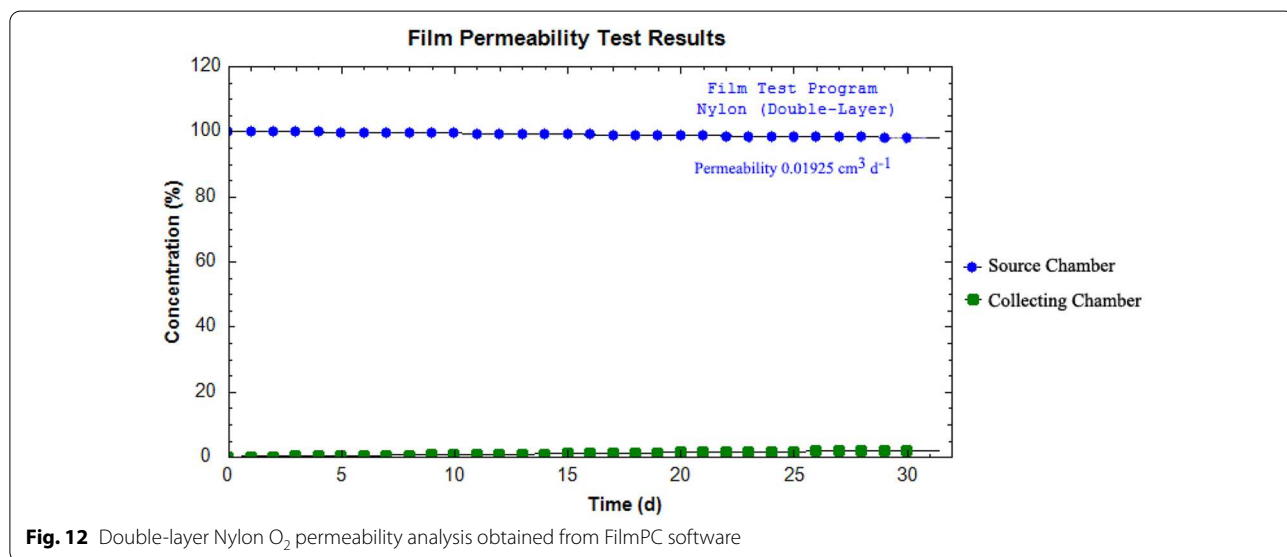
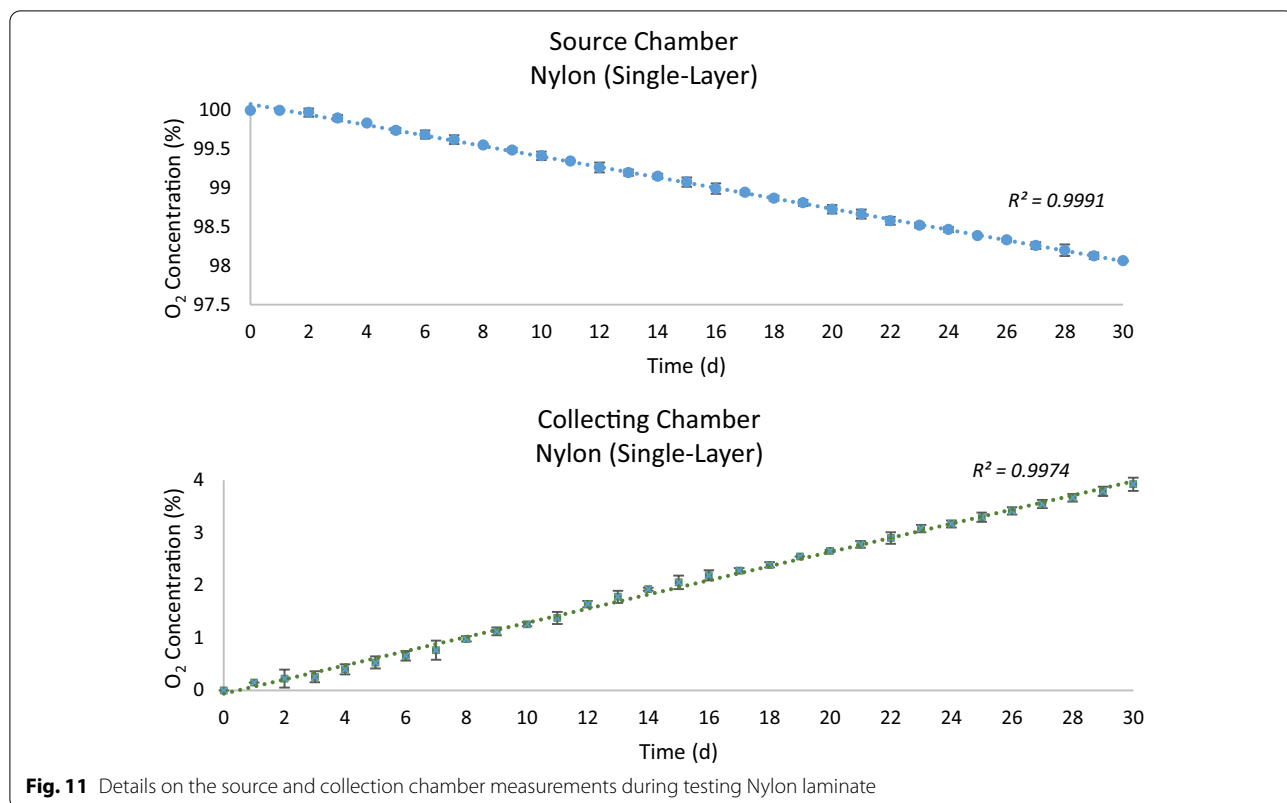




system. Therefore, one must make sure that the whole system (materials, connections, joints, paths, etc.) is leak-proof. For precise data analysis, experiments should be iterated.

**Conclusion**

The selection of consumable laminates is the main challenge when applying MIA treatments as they should meet some criteria to guarantee successful treatments

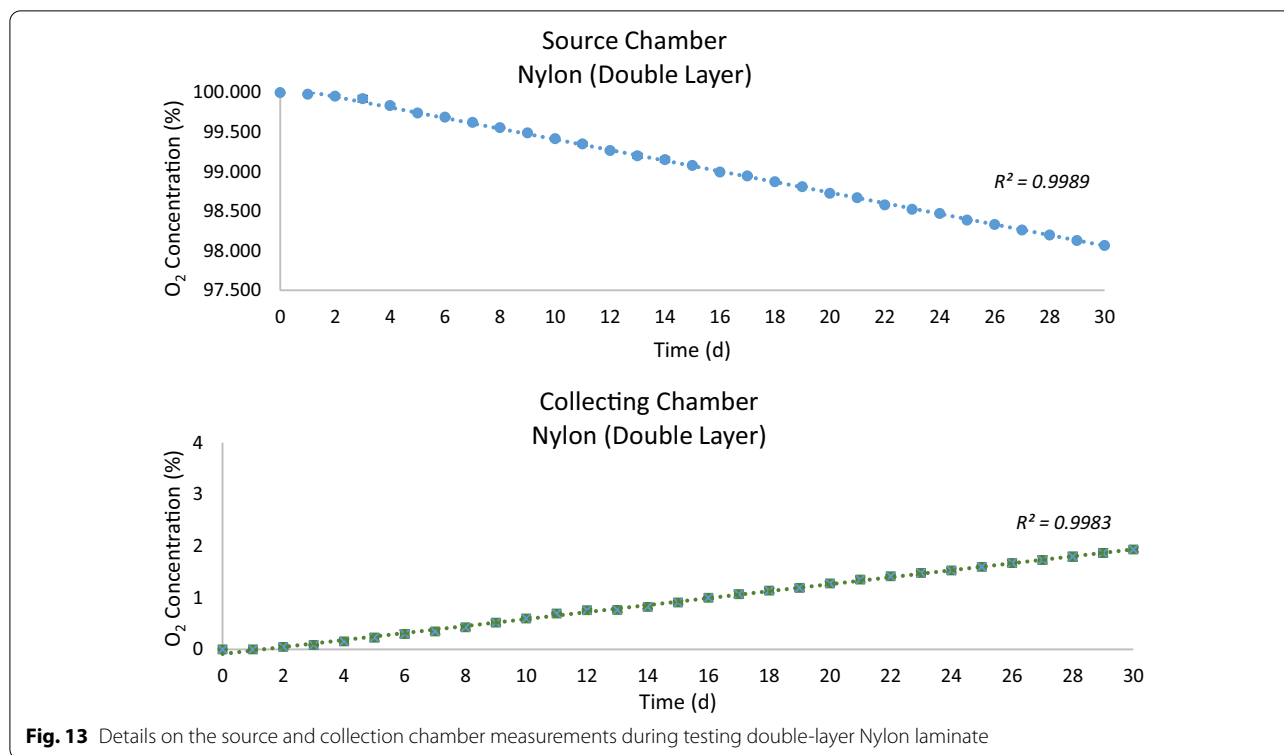


and be at acceptable costs to ensure sustainable application of the treatment, especially in limited-budget museums around the world.

In this study, a methodological approach adopted from the ASTM E2945-14 [15, 16] was developed to measure the oxygen permeability, and consequently, the OTR,

of film laminates. The developed test system allows for an unlimited number of tests on an unlimited number of laminates using materials and equipment normally available in MIA laboratories, such as oxygen sensors, leak-proof fittings, and glass or thick plexiglass. The system allows for easy consistent, and time-managed OTR





**Table 1** Properties of tested laminated films. Price is given three grades for comparison

Film Laminate	Manufacturer	Composition	Thickness (mm)	OTR (cm <sup>3</sup> m <sup>-2</sup> d <sup>-1</sup> )		Price /m <sup>2</sup>
				Spec	Meas	
ESCAL™	Mitsubishi Gas Chemical Co., Japan	(SiO) <sub>x</sub> / PET PE	0.11–0.12	< 0.1 [7] 0.05[12]	0.06	Very high
Cross-Barrier	Nissin Kasei Co., Ltd., Japan	(AlO) <sub>x</sub> / PET PE LLDP	0.14	NA	2.0	Moderate
Nylon	M2Pack Co., Cairo, Egypt	PA PE	0.092	NA	4.1	Low
Double-Layer Nylon		Alternate PA/PE	0.184	NA	1.9	Low

testing in museum laboratories. It also facilitates the selection among different laminates for MIA treatments to manage the cost and enhance the sustainability of their application and hereby accomplishes the main aim of this study.

Three different laminates were tested in this study. Results showed that ESCAL™ laminate, which is based on silicon dioxide, has the highest oxygen-barrier ability. Cross-Barrier, which is based on aluminum oxide, has almost the same OTR as double-layered Nylon, which is based on alternate polyamide/polyethylene layers. Although Nylon by default has the lowest OTR

compared to other tested laminates, the tested local type showed high oxygen barrier properties which adds to its improved flexibility, due to its thickness and lack of strengthening layers. Furthermore, its competitive price and local availability make it a suitable choice for wide-range, sustainable MIA applications in limited-budget museums worldwide.

**Abbreviations**

MIA: Modified inert atmosphere; OTR: Oxygen transmission rate; ASTM: International standards organization, formerly American Society for Testing and Materials; PVAL: Polyvinyl alcohol; OPP: Orientated polypropylene; LLDPE: Linear low-density polyethylene; PE: Polyethylene.

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### Authors' contributions

Conceptualization, methodology, experiment, validation, analysis, discussions and writing- ME, supervision, writing-review and editing-NE. Both authors read and approved the final manuscript.

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Not applicable.

### Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

### Declarations

#### Competing interests

The authors declare that they have no competing interests.

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