



Methods for gas permeability measurement in edible films for fruits and vegetables: a review

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Abstract

Permeability is a crucial parameter to describe the transport rate of a gas under pressure gradient, relevant to gas molecule movement in pores. The gas permeability of edible film, used on fresh or minimally processed fruits and vegetables, is an important parameter because it directly affects the product's respiration rate and metabolic activity. The present study summarizes the principal methods for gas permeability measurement in edible films, considering methods developed at laboratory scale and the methods of the American Society for Testing and Materials Standards (ASTM), some of which have been adapted for edible films. The methods are classified as follows: gravimetric, differential pressure, and continuous flow. Gas permeability results depend heavily on the procedure achieved and laboratory conditions employed, as well as the preconditioning of the films before the tests.

Keywords: respiration rate; permeation; film properties; postharvest.

Practical Application: Principal methods of gas permeability measurement for edible produce film and can serve as a guide to the selection of a proper method for gas permeability measurement on a laboratory scale.

1 Introduction

Edible films can be defined as a primary packaging material that is made of edible components, and can be obtained from casting or through thermo-formation for the subsequent coating of a given food surface (Falguera et al., 2011). Edible film has been used to provide moisture barriers on product surfaces, which decreases product weight loss. Also, it helps to control the gas exchange (i.e., decreased respiration, delay of enzymatic oxidation) and the volatile compound exchange between fresh products and the outside atmosphere (Khaliq et al., 2016).

Many investigations that study the shelf life of fresh, or minimally processed fruits and vegetables, using edible film, have been performed in recent years. This film has been applied to fruits, such as oranges (Youssef et al., 2015), tomatoes (Robledo et al., 2018), and grapes (Baraiya et al., 2016), with satisfactory results. However, the results are not always favorable, and can affect fruit quality, due to the modification of their internal atmosphere. This, in turn, causes alterations to the respiration and internal composition of the gases within the product (Ramirez et al., 2015).

Most recent investigations have focused primarily on the development of new films and the study of the properties (e.g. firmness, color, product weight loss) acquired by the food after film application (Kaya et al., 2018; Robledo et al., 2018; Zhang et al., 2019b). However, further studies are required to investigate both barrier properties and gas permeability, as well as to unify the procedures involved in the measurement of these parameters. The aim of this paper is to review the methods used to measure water vapor, oxygen, and carbon dioxide permeability in edible produce films.

2 Edible films

Edible films should meet specific requirements that depend on the type of product to be coated and the metabolic routes involved. These properties include adequate water vapor and solute permeabilities, selective permeability to gas and volatile compounds, uniform thickness, and being free of wrinkles, creases, pinholes, and other imperfections (Dhall, 2013). Edible films are generally formed from polysaccharides, proteins, lipids, or a combination of these compounds. Material barrier properties, concerning water vapor, oxygen, carbon dioxide, and the transfer of lipids, in specific food systems, depend on the compounds used (Dhall, 2013). Coating material selection is generally based on water solubility, hydrophobicity, ease of film formation, and sensory properties (Lin & Zhao, 2007).

3 Edible film permeability

3.1 Gas transmission rate

The Gas Transmission Rate (GTR) is the quantity of a given gas that passes through a unit of surface area that is perpendicular to a plastic film, per unit time, under given test conditions ($\text{mol}/\text{m}^2\text{s}$). Test conditions include a fixed temperature and partial pressure for the gas (American Society for Testing Materials, 2010).

3.2 Water vapor transmission rate

The Water vapor Transmission Rate (WVTR) is the quantity of vapor transferred through a material with a given area, during

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a given time ($\text{g/m}^2\text{s}$), under constant humidity and temperature conditions (American Society for Testing Materials, 2013).

3.3 Permeation and permeability

Permeation is the transmission rate of a gas (or water vapor), based on the difference in the partial pressure of said gas on both sides of a film, in $\text{mol}/(\text{m}^2\text{sPa})$ (American Society for Testing Materials, 2010). Figure 1 shows the sorption and diffusion mechanisms that occur on each side of the film, as well as through the film, during the permeation process.

The permeation process involves three steps. First, the permeated molecule, in the fluid phase, at partial pressure p_1 , penetrates the polymer surface. For pressures lower than one atmosphere, the permeate concentration of the polymer interface follows Henry's Law. Second, the permeate is diffused through the polymer film, from the side with a high concentration to the side with a low concentration, as in Fick's Law. Third, the permeate leaves the opposite polymer interface to diffuse into an adjacent continuous phase (i.e., gas or liquid), at pressure p_2 . Based on previous information, permeation can be mathematically described by Fick's first law.

The flux (J), which is proportional to the gradient of concentration, can be defined by Equation 1 (Geankoplis, 1993).

$$J = -D \left(\frac{\delta C}{\delta X} \right) \quad (1)$$

where J is the flux, or net quantity of solute, diffused through a unit area, per unit time ($\text{g}/\text{m}^2\text{s}$ or $\text{mL}/\text{m}^2\text{s}$); D is the diffusion constant (m^2/s); C is the concentration of the diffused substance, and X is film thickness (m).

The flux is described by Equation 2, where the following assumptions are made: first, diffusion occurs in a stationary state, and second, a linear gradient is present in the film.

$$J = \frac{D(C_2 - C_1)}{X} = \frac{Q}{At} \quad (2)$$

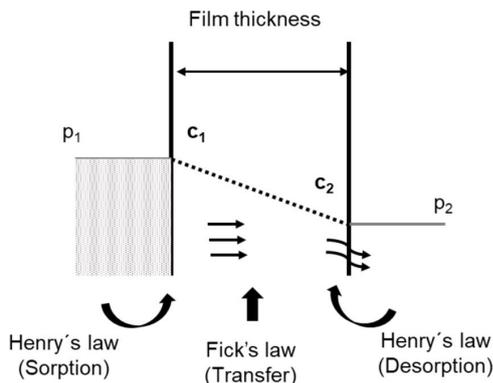


Figure 1. Permeation mechanisms through a film. Adapted from Valentas et al. (1997) Where p_1 is the partial pressure of gas molecules with concentration c_1 and, p_2 is the partial pressure of permeated gas on the opposite side of the film with a concentration c_2 .

where Q is the quantity of gas diffused through the film, in g or mL; A is the exposed area of the film (m^2); and t is the time (s) during which the process occurs.

Considering that the permeate concentration at the polymer interface follows Henry's Law (Equation 3) and the definition of permeability (Equation 4), Equation 5 may be obtained.

$$C_i = Sp_i \quad (3)$$

$$P = DS \quad (4)$$

where S is the solubility constant (mol/Pa); p_i is the partial pressures on each side of the film (Pa); and P is the permeability P ($\text{g}\cdot\text{m}/\text{m}^2\cdot\text{s}\cdot\text{Pa}$).

The flux (J) can be expressed by Equation 5:

$$J = \frac{D(C_2 - C_1)}{X} = \frac{DS(p_2 - p_1)}{X} = \frac{P(p_2 - p_1)}{X} \quad (5)$$

Replacing J in Equation 2, Equation 6 may be obtained:

$$\frac{Q}{At} = \frac{P\Delta p}{X} \quad (6)$$

By rearranging Equation 6, permeability can be expressed as:

$$P = \frac{QX}{At\Delta p} \quad (7)$$

Since permeability is the product of permeation and film thickness, dimensional analysis of permeation indicates that the fundamental units are $\text{g}/\text{s}\cdot\text{m}^2 \text{Pa}$ (Valentas et al., 1997).

4 Methods of measuring gas permeability in edible films

Generally, the methods used to estimate film permeability measure a signal that is associated with the quantity or transmission rate of a gas or vapor that has permeated the film. Most methods use the same principle: a measurement of the gas transmission rate through an edible film located between two compartments. One side of the film is exposed to the gas or vapor being studied (i.e., the food side), and a detector is placed in the other compartment, which is initially free of the permeated compound (i.e., the permeated side) (Martinez & Artés, 2005).

The ASTM provides methods to test water vapor transmission rates, as well as different methods of O_2 and CO_2 gas transmission measurement: the manometric method, volumetric method, and continuous-flow method for plastic film, sheeting, laminates, coextrusions, or fabrics. The first two methods measure the difference in absolute pressure, and the others use a stream flux of the gas to be measured on one side of the film and a nitrogen stream on the other side to carry the gas to the analyzer. Coulometric sensors, infrared sensors, or gas chromatography may be used for gas concentrations analysis.

Among the methods employed to measure gas permeability for edible films and those established by ASTM standard test methods, different variations, which depend on the method used to determine the transmission rate of the gas that permeated

the film can be utilized. Based on previous information, the current methods applied to measure the gas and water vapor permeability of films and their adaptation to edible films have been classified and studied. Figure 2 shows the classification of the methods used to measure gas permeability through these types of films.

4.1 Gravimetric and static methods

Water vapor transmission rate

The measurement of the water vapor permeability transmission rate is determined by the ASTM E96 (American Society for Testing Materials, 2011) and ASTM D1653 (American Society for Testing Materials, 2013). Both standards rely on two primary methods: the desiccant and moist method. The selected method depends on that which best suits the film's operational conditions.

The graph of weight gain, as a function of time, determines the water vapor transmission rate. Said rate corresponds to the slope of the linear part of the graph, as a stationary state is assumed. With both methods, the water vapor transmission rate is calculated with Equation 2. Frequently the flux (J) is called the WVTR (Water Vapor Transmission Rate) ($g/s\cdot m^2$).

Water vapor permeability may be determined with Equation 8, considering that partial pressure is the relative humidity, multiplied by the saturation pressure:

$$P = \frac{(WVTR)(X)}{\Delta p} = \frac{(WVTR)(X)}{p(RH_1 - RH_2)} \tag{8}$$

where Δp is the vapor pressure difference in Pa; p is the saturation vapor pressure at the test temperature in Pa; RH_1 is the relative humidity of the source, expressed as a fraction (i.e., the test chamber with the desiccant method and the container with the water method), and RH_2 is the relative humidity of the vapor output.

There are instruments on the market that purport to measure the water vapor transmission in films more easily and rapidly than the methods described in test methods D1653 and E96 (e.g., The water vapor transmission system developed by LabThink) (Labthink, 2016). According to the ASTM D1653, this type of system runs primarily the same kinds of tests as in ASTM methods, but does so instrumentally (American Society for Testing Materials, 2013).

Numerous studies have reported the Water Vapor Permeability (WVP) of edible films, based on ASTM E96 (American Society for Testing Materials, 2011). Ballesteros-Mártinez et al. (2020) determined the WVP of sweet potato starch films, at 22 °C, using a test cup containing distilled water and placed the test cups in a desiccator containing silica gel (0%HRH). Assis et al. (2018) determined the WVP of cassava starch films using test cups containing granular anhydrous calcium chloride (0% RH) and stored them in a glass chamber with saturated sodium chloride solution (75% RH).

Oxygen and carbon dioxide transmission rates

Ayranci et al. (1999) developed a method to measure carbon-dioxide transmission through edible films that did not use flux or gas analyzers. This method is an adaptation of the static method for the measurement of water vapor transmission described by the ASTM E96 (American Society for Testing Materials, 2011), with modifications for carbon dioxide. With this method, the transferred quantity of CO₂ is determined by the weight gain of a solid adsorbent (ascarite II), which consists of calcium chloride in a non-fibrous silicate that retains carbon dioxide. The assembly scheme used in this method is as shown in Figure 3.

Zhang et al. (2019c) reported the use of gravimeter methods to measure the rate of oxygen and carbon dioxide transmission in edible film. The oxygen transmission rate was measured by deoxidizer absorption, and the carbon dioxide transmission rate was measured using the KOH absorption method. For both methods, the authors placed an absorber (deoxidizer or KOH) into a glass cup. Subsequently, they were placed in a constant temperature and humidity chamber (23 °C, RH 90%) for 48 hours. The deoxidizer adsorbs O₂ through the coatings or, the KOH adsorbs CO₂. The gas transmission rate is calculated using Equation 2.

4.2 Methods of pressure measurement

Pressure differential methods are used for the measurement to N₂, O₂, and CO₂ permeabilities in edible films. The method uses a pressure difference between the two sides of the sample under study. This pressure difference is the driving force for permeation. With this method, the gas being studied passes

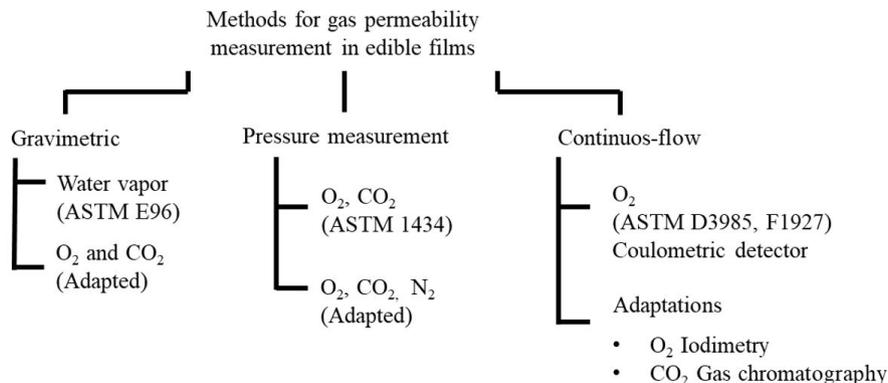


Figure 2. Methods for gas permeability measurement in edible films.

through the film and accumulates in a vacuum-sealed chamber of a known volume. The increase in pressure on this side, where the pressure is lower, is detected by a high-precision pressure sensor, which allows the permeation to be calculated. The amount of permeate gas is calculated using the Equation 9 of ideal gases:

$$N_i = \frac{P_T V}{RT} \quad (9)$$

where N_i is the amount of gas (mol); V is the volume of the permeated gas (m^3); P_T is the total pressure in that volume (Pa); R is the universal constant of gases ($\text{m}^3\text{-Pa}/(\text{mol}\cdot\text{K})$); and T is the temperature of the gas (K). With the amount of permeated gas, permeability can be calculated using Equation 7, where Δp corresponds to the pressure in the lower and upper sides of the chambers.

The ASTM D1434 (American Society for Testing Materials, 2009) standard describes ways to determine gas permeability using this method. In this standard one of the chambers contains the test gas, at a specific pressure, and the other receives the permeated gas, at low pressure. An increase in pressure indicates that gas is being transmitted through the film. Using the ASTM D1434 estimated semi-quantitative results are obtained for the pure-gas transmission rate through the films. Therefore, this method indicates that the correlation of the values measured for a given packaging application should also be determined by experience. Furthermore, the transmission rate is affected by conditions unspecified by this method, such as relative humidity or the plasticizer utilized. The permeability tests performed with this method depend heavily on the procedure implemented and the laboratory conditions used. Therefore, congruity with other methods is poor, and depends upon the material (Schmid et al., 2015).

4.3 Continuous-flow or isostatic method

The apparatus used to determine gas permeability via the isostatic method, consist of two or three chambers, separated

by a film, at atmospheric pressure. Gas transmission through a film depends upon the partial pressure difference on each side of the film, not on the total pressure difference (Martinez & Artés, 2005). The overall system used for this type of method is shown in Figure 4. A cylindrical device is separated into two cells by a film with a known thickness and area. The gas (i.e. O_2 or CO_2) flows through one of the cells, while N_2 flows through the other as a carrier gas. The gas being studied permeates the film and is carried by the N_2 to a flux meter, where the total flux is determined and subsequently passed to the gas detector, which measures the concentration of the permeated gas in the mixture (Vargas et al., 2007).

Permeability is calculated using Equation 7, where the partial pressure difference is obtained by considering the partial pressure of a gas to be equal to its molar fraction, multiplied by total mixture pressure. Because one cell contains 100% of the permeate and the other contains 0% of the permeated gas, their pressures are, respectively, 1 and 0 atm., and Δp is 1 atm. Cissé et al. (2012) used a standard isostatic method for the oxygen and carbon dioxide permeability measurements in chitosan-based edible films, and Guillaume et al. (2013) assessed the oxygen permeation of wheat-gluten-coated paper in accordance with an isostatic method. The gas detectors used in these studies were gas sensors or gas chromatographs.

Electrochemical methods (i.e. coulometry)

Coulometric methods of analysis are based on the exact measurement of the quantity of electricity that passes through a solution during an electrochemical reaction. The sensor detects this reaction as an electrical current, with a magnitude that is proportional to the amount of oxygen that flows to the sensor, per unit time. Each oxygen molecule that enters the sensor is analyzed, allowing the measurement of the Oxygen Transmission Rate (OTR) with an efficiency of 95-98% (Liptak, 1994). Two ASTM standard methods are available to analyze oxygen transmission rates, using coulometric sensors: ASTM

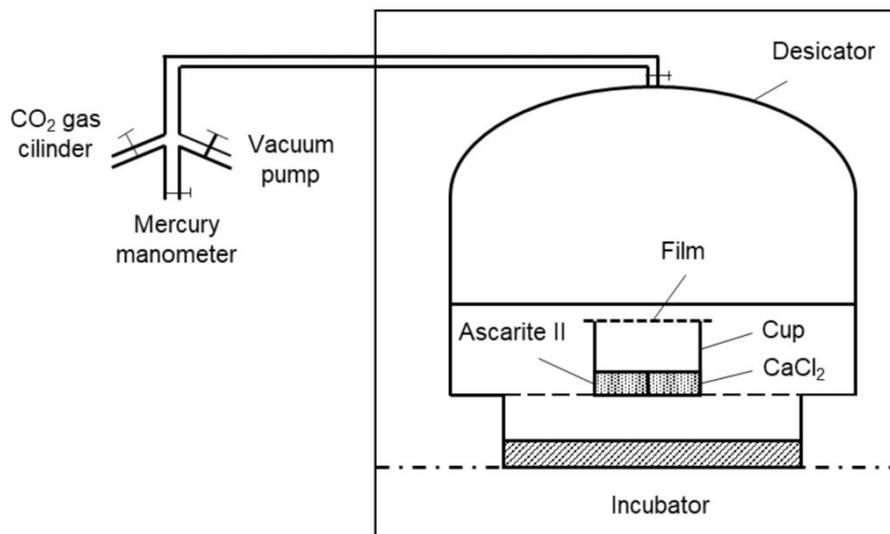


Figure 3. Scheme of the apparatus used for CO_2 transmission measurement (Ayranci et al., 1999).

D3985 (American Society for Testing Materials, 2010) and ASTM F1927 (American Society for Testing Materials, 2014).

In the ASTM D3985 the film studied acts as a semi-barrier between two chambers at atmospheric pressure. One of the chambers is slowly purged with a nitrogen stream, and the other chamber contains oxygen. As the oxygen permeates the film into the nitrogen chamber, it is transported to the coulometric detector, where an electric current is produced. This method is only applied in dry conditions, and the oxygen gas transmission rate is determined after the sample has equilibrated in a dry test environment (RH less than 1%). This last condition could be inconvenient for edible films, as they can become brittle or porous in very low humidity environments.

The ASTM F1927-14 (American Society for Testing Materials, 2014) describes a method that uses the same principle as that described in the ASTM D3985, except that it allows the tests to be performed at different relative humidities. This is an important distinction because the permeation rate of different materials is affected by relative humidity, which causes changes in the oxygen transmission rate.

Chemical methods (i.e. iodimetry)

This method was designed by Ayranci & Tunc (2003) to measure the oxygen permeability of edible film, and is based on the method described in the ASTM D3985 (American Society for Testing Materials, 2010) standard. This modification allows for the analysis of O₂, via a simple wet chemical analysis, based on iodometry (Figure 5). This is used to determine the quantity of permeated oxygen, instead of using an oxygen analyzer. The permeated mixture of N₂ and O₂ passes through the wet system for a known period and is analyzed for O₂.

4.4 Methods and test conditions for gas permeability measurement in edible films

Table 1 compares methods and test conditions for gas and water vapor permeability measurement in edible film. The wide variety of equipment used for this type of measurement confirms the complexity of the procedure.

For O₂ permeability it was found that most of the authors used the continuous flow method, based on ASTM D3985

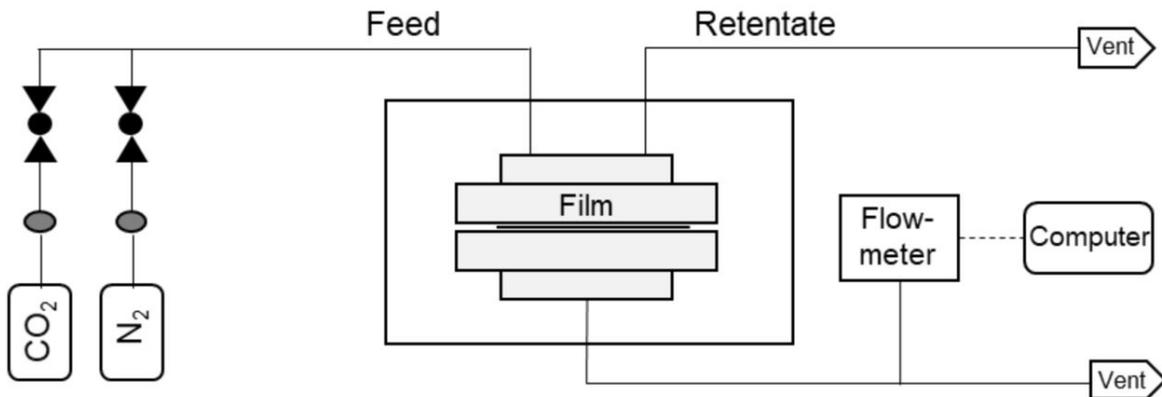


Figure 4. Continuous flow method system used to measure CO₂ gas permeability. Adapted from Tan et al. (2015).

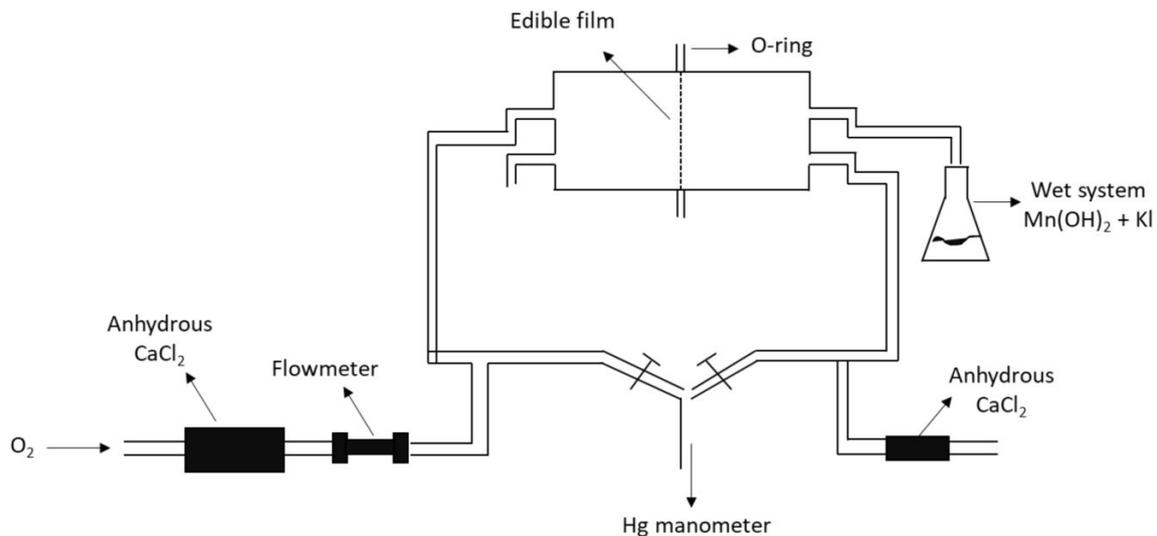


Figure 5. Setup of the equipment used to measure the O₂ edible film permeability. Adapted from Ayranci & Tunc (2003).

Table 1. Methods for gas and water vapor permeability measurements in edible films.

Edible film	Gas	Method/Equipment	Test condition	Films Precondition	Reference
Sago starch with cinnamon essential oil and nano TiO ₂	O ₂	ASTM D3985-17 Mocon Oxtran 2/21 System	Not reported	25 °C, 55% RH, for 48 h	Arezoo et al. (2020)
Whey protein with almond and walnut oils	O ₂	OXTRAN 2/21 MH modular system. ASTM F 1927-98	23 °C, 50% RH	25 °C, 53% RH for 48 h	Galus & Kadzińska (2016)
Zein/chitosan	O ₂	Deoxidizer absorption method	23 °C, 75% RH	Not reported	Zhang et al. (2019a)
Quince seed mucilage with oregano essential oil	O ₂	A Gas Permeability Tester (GDP-C- D-80335). ASTM D3985	23 °C, 59% RH	25 °C, 50% RH for 48 h	Jouki et al. (2014)
Locust bean gum with whey protein	O ₂	Based on ASTM D3985-02. O ₂ P were determined by gas chromatography	Not reported	25 °C, 53% RH for one week	Silva et al. (2016)
Salep mucilage with pennyroyal	O ₂	Gas permeability tester based on ASTM D3985	25 °C, 53% RH	Not reported	Ekrami et al. (2019)
Citrus pectin with transglutaminas	O ₂	MultiPerm apparatus. Modification of ASTM D3985-8129	25 °C, 50% RH	50% RH For 48 h	Giosafatto et al. (2014)
Whey protein isolate	O ₂	Oxygen-specific carrier gas method (continuous flow method).	35, 50 and 75% RH	Not reported	Schmid et al. (2015)
Konjac glucomannan/Carrageenan/nano-SiO ₂	O ₂	Deoxidizer absorption method. (Gravimetric method)	23 °C, RH 90%. 48 h	Not reported	Zhang et al. (2019c)
Hydroxypropyl methylcellulose films with curcumin polymorphs	CO ₂	ASTM D1434, apparatus assembled for permeability measurement	25 °C	Not reported	Nascimento da Silva et al. (2019)
Whey protein with almond and walnut oils	CO ₂	A Permatran-C 4/41 with an infrared detector. ASTM F 2476-05	23 °C	25 °C, 53% RH for 48 h	Galus & Kadzińska (2016)
Locust bean gum with whey protein	CO ₂	Based on ASTM D3985-02. CO ₂ P were determined by gas chromatography	Not reported	25 °C, 53% RH for one week	Silva et al. (2016)
Zein/chitosan	CO ₂	Alkali absorption method	23 °C, 75% RH	Not reported	Zhang et al. (2019a)
Citrus pectin with transglutaminas	CO ₂	MultiPerm apparatus Modification of ASTM D3985-8129	25 °C, 50% RH	50% RH For 48 h	Giosafatto et al. (2014)
Whey protein isolate	CO ₂	Manometric method according to DIN 53380-2. Absolute pressure difference	25 °C	Not reported	Schmid et al. (2015)
konjac glucomannan/carrageenan/nano-SiO ₂	CO ₂	KOH absorption method (gravimetric)	23 °C, RH 90%. 48 h	23 °C, 50% RH for 24 h	Zhang et al. (2019c)
Citrus peel low-methylated	CO ₂	MultiPerm apparatus A modified ASTM D3985-81 (1981)	25 °C, 50% RH	25 °C, 53% RH for 24 h	Esposito et al. (2016)
Pectin from apple and strawberry	Water Vapor	Gravimetrically. ASTM E96-00	25 °C, RH gradient (33-65%)	25 °C, 58% RH for 48 h	Eça et al. (2015)
Konjac glucomannan with bacterial cellulose nanofibers	Water Vapor	Gravimetrically	20 °C, 80% RH	Not reported	Liu et al. (2020)
Sweet potato starch, glycerol, sorbitol	Water vapor	Gravimetrically. ASTM E96-80	22 °C, 0% RH	Not reported	Ballesteros-Márquez et al. (2020)
Locust bean gum with whey protein	Water Vapor	Gravimetrically. ASTM E96-95	25 °C, RH gradient (2-100%).	25 °C, 53% RH for one week	Silva et al. (2016)

(American Society for Testing Materials, 2010) with variations in test conditions, while only Zhang et al. (2019c) reported having used the gravimetric method. For the CO₂ permeability measurements of edible film, a higher number of methods and adaptations were found. These included continuous flow, gravimetric methods, and gas chromatography. For all cases, differences in temperature and relative humidity conditions were found.

One of the most commonly-reported methods in Table 1 is based on the ASTM D3985 (American Society for Testing Materials, 2010) or its adaptations. The adaptations for this method consist mainly of replacing the coulometric sensor with CO₂ infrared sensors, or the detection of permeated gas using gas chromatography. The ASTM 3985 standard is for dry conditions only, and the temperature must be monitored, as it is a critical parameter. Also, the oxygen gas transmission rate should be determined after the sample has equilibrated in a dry test environment (RH<1%). For this last condition, it is essential to mention that drying edible film for long periods of time, at low relative humidity, can cause their deterioration, making them brittle or porous (Schmid et al., 2015). Table 1 also shows that Galus & Kadzińska (2016) reported the use of the ASTM F1927 (American Society for Testing Materials, 2014) for O₂ permeability measurements, which can be an appropriate alternative for edible film, because it allows for relative humidity variations.

For water vapor permeability, Table 1 shows that all measurements are based on the ASTM E96 (American Society for Testing Materials, 2011), and water vapor permeability values have been reported at different relative humidity gradients. This method also requires long-duration measurements, which can stretch from eight to 48 hours.

As stated by the ASTM E96 method, a permeance value obtained under one set of test conditions may not indicate the value under a different set of conditions. Further, test conditions should be selected to most closely approach the conditions of use. For the investigations reported in Table 1, it is not known whether the conditions used were selected for the possible future use of the films, or if they correspond to the conditions that could be evaluated in the laboratory. In all cases, the water vapor permeability reported is not comparable to other similar investigations.

5 Conclusions

The static or gravimetric method for water vapor permeability can be used as an inexpensive alternative on a laboratory scale. However, it is necessary to standardize relative humidity and temperature conditions for film sample preparation.

The methods of pressure difference for oxygen and carbon dioxide permeability have advantages, as compared to chemical methods, as edible films with different compositions can be measured, and it is also possible to adjust the temperature and vacuum pressure conditions for each test. However, for this method, specific mechanical film properties are required. Otherwise, they are affected by the vacuum conditions mentioned in the ASTM standard for plastic film.

Edible film may become brittle when subjected to the conditions established by the permeability measurement methods, such as low relative humidity, vacuum pressures, or a constant gas (i.e., O₂, CO₂, water vapor) flow for a prolonged time. These challenges should increase the development of new methods or the adaptation of existing methods with lower operational costs and adaptabilities for edible film.

There is no standard technique to measure the gas permeability of edible films that allows for the reliable reporting and comparison of results obtained from investigations. The development and standardization of methods for measuring these barrier properties have great importance for the food packaging industry, and especially for the commercialization of fresh or minimally processed produce. Efforts should focus primarily on controlling factors that affect the transmission of water vapor and gases, such as temperature and relative humidity, as well as factors such as vacuum pressure and drying conditions for film sample preparation.

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