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Measurement of Permeability of Food Packaging Polymer Films to Organic Vapors

– Review –

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Abstract

The need to determine the permeability of organic vapors to polymeric films such as aromas, flavors, etc. has significantly increased in the food industry because of preservation and safety issues along with migration problems. However, due to the complex nature of the permeation process, there have been few investigations compared to non-organic compounds. In this paper, we review the history of permeability studies and typical methods of permeability measurement such as the isostatic method and quasi-isostatic method for organic vapors. New instrumental developments and significant findings are also introduced and discussed.

Key words: permeability, food packaging, polymer film, organic vapor

INTRODUCTION

Plastics are very versatile packaging materials and now used in 40% of all packaging. They are lightweight, cheap, strong, and easily convertible and now widely used for packaging foods, cosmetics and perfumed household products. Especially in food packaging, a large number of polymeric materials are used. Oxygen, moisture vapor and aroma barrier properties are significant factors in the choice of food packaging materials (1).

As the demands on barrier plastics are growing, the necessity to understand the transport properties of polymeric packaging films, regarding oxygen, moisture, organic vapors, aromas and solvents has been created although data is still lacking on the last three of these. The trend to replace glass and metal packages with plastics emphasizes the necessity for protection of flavor, aroma and solvents, without product additives. Organic vapors such as flavors must remain within the package, and no flavors or aromas should permeate from outside. Flavor scalping, absorption of flavors from the product by the plastic packaging, and desorption of flavors in the reverse way, must be avoided. Solvents from printing inks, for instance, toluene, with reverse printed films, must not diffuse into the food, while solvents from packaged chemicals and pharmaceuticals must not escape (2,3).

Today, consumers are much more demanding and want food that is perfectly safe, nutritious and tasty. However, there are no plastics that are impermeable. If a packaging designer wants a plastic material that is to protect a food product from external odors, maintain its original flavor and ensure an adequate shelf life by protecting flavors of the food from diffusion outwards through the packages, it is necessary to understand its performance characteristics with respect to organic compounds.

Factors affecting permeability of organic vapors relate to the polymer used, the aroma itself, external parameters and the packaged product. A polymer's barrier properties are related to the size of its constituent molecules and the force binding them. Factors, which affect organic vapors, are its type (natural, naturally simulated, synthetic), volatility, molecule shape and size, polarity and mixture. The main external factors are temperature and humidity while product parameters include pH level, water and lipid content, rheological characteristics and the substances of the flavor (4).

In this paper, we will review the history of the permeability studies, theories, and typical instrumental methods for organic vapors. Hence the history of organic vapors studies is relatively short and developed instrumental technologies are fairly new compared to water and inorganic compounds, and there is still much room for revealing permeation behaviors of organic vapors.

HISTORY OF PERMEATION STUDIES

Organic vapors, volatile low-molecular weight organic compounds, are important constituents of foods, because of their influence on the characteristic flavor of a food. In general, protection against loss of the aroma constituents from foods is achieved by the use of appropriate packaging. Glass and metal are excellent barrier materials, however, increasing demands on polymeric materials prompted the development of new permeability studies.

Most permeation studies for organic vapors are due to new development of barrier films, for example, EVOH, Nylon, PET, PVDC, OPP, etc., which have aroused great interest. Organic vapor barrier may be tested using the gas permeation

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method or by sensory evaluation testing. Some highly interesting and relevant work has been done on instrumental methodology. Allison (5) found that Nylon has a better aroma barrier than ethylene vinyl alcohol copolymer, and metalized films and PVDC-coated materials also have good aroma barriers.

The performance of PET bottles as alternative containers for wine compared to the traditional glass bottle was examined at the National Food Industries Instruction Wine Center in Japan. The results showed that oxidizing odors and aroma deterioration speed is higher in PET bottles giving the product a commercial life of only six to eight months or about half that of wine in glass bottles (6).

Mobil research center (7) in Virton, Belgium, tested the ability of acrylic and PVDC-coated OPP films to seal in aromas with toluene and limonene which were used as simulants. Results showed that metalized lacquered foil laminates such as Mobil Plastics Europe's Metallyte products offer better aroma protection than metalized coextruded laminated films. Lindner-Steinert (8) compared the aroma permeability of aluminum-paper laminate, metalized paper and printed paper cigarette inner wraps, using 7 aroma agents in polyethylene glycol solution. Relative aroma permeabilities were found to be similar to water vapor permeabilities. Aluminum-paper laminate provided the best barrier, followed by metalized paper, and printed-paper had the highest permeability. Tests on the entire package (inner wrap, carton and polypropylene overwrap) gave similar results. Aroma absorption by the carton was determined by inner wrap permeability. Lindner-Steinert (9) also compared the effect on coffee aroma of four packaging materials : 12 μm PET/7 μm Al foil/70 μm PE, metalized 12 μm PET/80 μm PE, 12 μm PET/80 μm PE/EVOH multilayer, and silicon oxide coated 12 μm PET/100 μm PE. Best aroma protection was provided by the aluminum foil laminate, while the metalized film structure showed the highest permeability rates.

In order to determine the permeability of a packaging material to aromas, it is not adequate to know its permeability to oxygen and water vapor and the partial pressure on the packaging material and the density of the product packed are also important. Dubois (10) used various aromas, limonene, menthol, garlic, onion and cassia as simulants for testing of different OPP films laminated with acrylics and PVDC and coextruded OPP films. The best barrier was provided by a dual PVDC and acrylic coated OPP laminate.

Extensive research, conducted by Mobil Plastics, indicates that the permeation rates of water vapor, oxygen, aromas and odors are not directly related. This research was conducted to find out the off-odor permeation and the aroma preservation of oriented polypropylene (OPP) films. The research concentrated on the action of these films as a barrier to odors, and whether certain films promoted a better retention of the flavor of the food. Results proved that aroma penetration is concentration dependent and that under both laboratory and normal conditions, coated films offer much better barrier properties than do uncoated films.

Coated OPP films were also shown to be more effective than coextruded films at keeping the aroma in the package. Until recently it was assumed that OPP films having a poor oxygen barrier would also tend to allow a high penetration of odors both from outside into the food and vice-versa. As the results illustrate, OPP materials coated with acrylic or polyvinylidene chloride (PVDC) provide an excellent barrier and also lead to better flavor retention (11,12). Similar research at Michigan State University's School of Packaging indicates that coated oriented polypropylene films serve as a barrier to flavors and odors, but the effectiveness of the barrier depends on the concentration of the permeating medium (13). At high concentrations, films coated with acrylic or PVDC were no more efficient than uncoated films, but at normal concentration levels they reportedly offer superior product protection for longer. The tests used toluene, ethyl acetate and limonene as simulants.

PERMEATION THEORY

Permeation is generally considered to be the movement of a material such as a gas or organic vapors from one side of a packaging material through to the other side of the package. In the actual world, the permeation occurs through a multi-step process. The permeant must collide with the polymer adsorbed to the surface, and then, be dissolved into the polymer. The permeant will move from the place where the concentration of its own kind is high to a place where the concentration is low. The mechanism of diffusion involves a series of random "jumps" from a free volume between polymer chains to another hole or void. Finally, the permeant desorbs from the surface and escapes into the environment (14). This means that permeation involves three steps : sorption at high concentration difference, diffusion through bulk phase, and desorption or evaporation from the surface.

The permeability is a measure of the overall migration rate of a permeant through a polymer, and sorption and diffusion rate determine permeability of a polymer. Hence, the permeability, P, as shown in Equation (1) is an important property of the polymer.

$$P = D \times S \quad (1)$$

where,

D = the diffusion coefficient, and
S = the solubility coefficient.

Sorption can also change the properties of polymer, which will have an effect on permeation, diffusion and additional sorption of compounds into the polymer, resulting in new barrier characteristics (15).

The basic equation to obtain diffusion value (D) is described from Fick's first and second laws.

$$F = -D\frac{dc}{dx} \quad (2)$$

$$\frac{dc}{dt} = \frac{d}{dx}\left(D\frac{dc}{dx}\right) \quad (3)$$

Where F is the flux (the rate of transfer of penetrant per unit area); D is the diffusion coefficient; c is the concentration of the penetrant in the film; t is time; and x is the length in the direction in which transport of the penetrant molecules occurs. If diffusion coefficient (D) is concentration dependent, the diffusion process is Fickian, but if D is time dependent, the diffusion process is non-Fickian (16).

However, when permeation involves interacting organic vapors, the relationship between P, D, S is more complex than that indicated by Equation (1) because of their swelling behavior into the film. In order to measure accurate permeation behavior of organic compounds, we need to determine the diffusion coefficient, solubility coefficient, and permeability constant. Diffusion coefficient (D) and permeability constant (P) can be found from two permeability measurements: by monitoring continually the transport of a permeant through a polymer membrane (isostatic method) or by quantifying the amount of the penetrant that has passed through the film and accumulated as a function of time (quasi-isostatic method) (17,18).

Isostatic method

At the isostatic method, $(\Delta M/\Delta t)_t$ at time t to ($\Delta M/\Delta$ value at the steady state is expressed as the equation below.

$$\frac{(\frac{\Delta M}{\Delta t})_t}{(\frac{\Delta M}{\Delta t})_\infty} = (\frac{4}{\sqrt{\pi}})(\frac{l^2}{4Dt})^{\frac{1}{2}} \exp(\frac{-l^2}{4Dt}) \tag{4}$$

Where, $(\Delta M/\Delta t)_t$ is the transmission rate of penetrant a time t, $(\Delta M/\Delta t)_\infty$ is the transmission rate of penetrant steady state; and l is the thickness of the film. A typical transmission rate profile curve for describing isostatic method is shown in Fig. 1. For each value of $(\Delta M/\Delta t)_t/(\Delta M/\Delta t)_\infty$, a va of $(l^2/4Dt)$ is calculated. When $(4Dt/l^2)$ is plotted as a function of time, a straight line is obtained. From the slope of this graph, D can be found by substitution in Equation (5).

$$D = \frac{(slope) \cdot l^2}{4} \tag{5}$$

Equation (4) is used to solve D.

$$D = \frac{l^2}{7.199 t_{0.5}} \tag{6}$$

where $t_{0.5}$ is the time required to reach a rate of transmission $(\Delta M/\Delta t)_t$ equal to half the steady state ($\Delta M/\Delta$ value.

The permeability coefficient $(\bar{P})$ can be determined from the isostatic method by substitution into Equation (7).

$$\bar{P} = \frac{a \cdot G \cdot f \cdot l}{A \cdot b} \tag{7}$$

where:

a=calibration factor to convert detector response to units of mass of permeant/unit of volume [(mass/volume) /signal units]

G=response units from detector output at steady state (signal units)

f=flow rate of sweep gas conveying penetrant to detector (volume/time)

A=area of the film exposed to permeant in the permeability cell (area units)

l=film thickness (thickness units)

b=driving force given by the concentration or partial pressure gradient (pressure or concentration units)

Quasi-isostatic method

A generalized transmission rate profile curve describing the transport of a permeant through a polymer membrane by the quasi-isostatic method is shown in Fig. 2. This method is to measure the permeated gas or vapor accumulated and total quantity of permeant to have transmitted through the film is plotted as a function of time. Barrer (19) showed a solution to determine diffusion coefficient (D) as follow Equation (8).

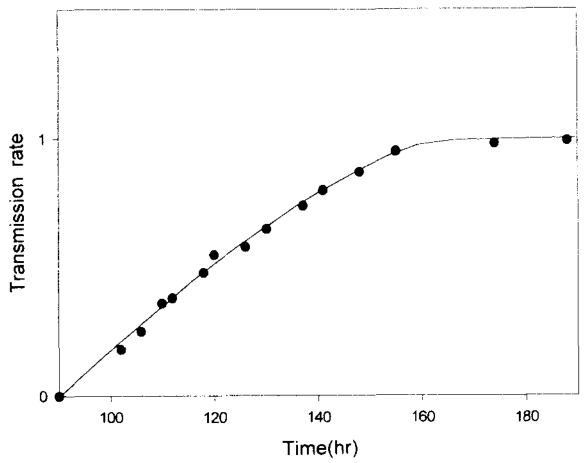

Fig. 1. Typical plot of transmission rate versus time for isostatic method of test.

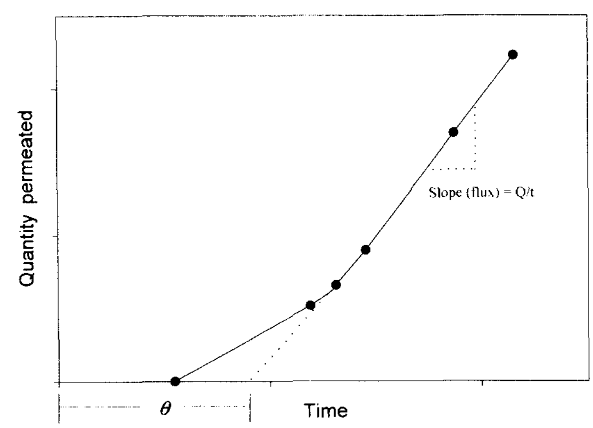

Fig. 2. Typical plot of transmission rate versus time for quasi-isostatic method of test.

$$D = \frac{l^2}{6\theta} \tag{8}$$

θ is lag time, the intermission of the projection of the steady state portion of the transmission curve and l is film thickness used for the test. Then, permeability constant can be obtained from quasi-isostatic method by substitution into Equation (9).

$$\bar{P} = \frac{y \cdot l}{A \cdot b} \tag{9}$$

where:

y=the slope of the straight line portion of the transmission rate curve (mass/time)
l=thickness of the film
A=area of the film exposed to the permeant in the permeability call
b=driving force given by the concentration or partial pressure gradient

INSTRUMENTAL DEVELOPMENT OF PERMEATION STUDIES

Although the permeation studies for organic vapors are fairly new compared to other non-organic compounds such as water vapor or oxygen, many different instruments have been applied for the studies. At the beginning of permeability studies for organic vapors, sensory evaluation is almost the only way to determine loss of flavors. The staling of roast coffee can be analytically measured by the determination of certain indicator substances that show good correlation with changes in sensory values (20). This study showed that the staling rate of coffee aroma was considerably affected by temperature, that ground coffee staled 5~10 times faster than whole beans, that vacuum packaging completely preserved coffee aroma for 6 months, and that interim storage time of 3 hours at a maximum with vacuum packs did not lead to any pre-deterioration of coffee aroma.

After major chemical companies such as Mobil pay attention to developing new barrier polymeric materials, most instrumental developments have been devised for the new material testing. Mobil Plastics Europe launched a marketing campaign for OPP film used to seal in the aromas of dried food products. For biscuits, peanuts, sweets and similar snack foods, a tight gas barrier is required. Considerable improvements have been made in the barrier properties of these films against oxygen, water vapor and carbon dioxide but the barrier properties of a film to flavor or aromas are not directly related to its oxygen barrier properties. The main part of the unit is a permeation cell consisting of a sealed airtight box divided into two separate compartments separated by a layer of test film. Nitrogen containing a fixed amount of the volatile aromatic component flows into one part of the cell and pure nitrogen flows into the other part. The equipment measures the time for gas transmission to take place (21).

A mass spectrometer is used to monitor d-limonene and trans-2-hexenal permeability through 2 barrier polymers, one a vinylidene chloride copolymer and the other an EVOH copolymer (1). High temperatures caused a large increase in permeabilities, and high humidity affected the EVOH copolymer.

A common method for measuring the gas permeabilities of packaging laminates is using flame-ionization detection of aroma molecules transmitted across a membrane in a two-compartment diffusion cell. The method was used to examine PE/paper and PVDC/paper laminates (22). Two different permeation mechanisms were found to exist, the mechanism involved in each case depended not only on the membrane material used and on the thickness of the plastic film layer, but also on the kind of permeating gas or vapor. The mechanism can be ascertained from the temperature dependence of the diffusion.

Permeability studies were also conducted using an isostatic procedure and the MAS Technology Model 2000 permeation test system (23). Tests for a series of organic vapor/polymer membrane combinations were set at three temperatures, with three vapor activity levels for each temperature. It was concluded that temperature strongly affects permeability and diffusivity of flavor and aroma compounds in barrier structures, concentration levels and also affected permeance values. The diffusion process was Fickian. Experimental values of temperature and vapor concentration were controlled and the MAS Technology test system was effective. The barrier properties of the tested films in order of decreasing barrier performance were metalized polyethylene terephthalate/ oriented polypropylene (OPP), acrylic coated OPP, polyvinylidene chloride copolymer coated OPP, OPP, high density polyethylene and glassine (23).

Apostolopoulos and Winters (24) developed a method to allow measurement of permeation for d-limonene at low concentrations, like those found in food packaging using permeation cells in conjunction with gas chromatography. The same method could determine the effect of temperature and relative humidity on permeation. Polyester (Mylar) and oriented polypropylene films were tested at very low vapor concentration gradients. The permeation behavior of packaging films at even lower levels, typical of foods, could be extrapolated, allowing more reliable estimation of flavor loss through the package, and of shelf-life (24). Gas chromatography has been traditionally used for testing barrier properties of resins such as ethylene vinyl alcohol copolymer (25).

Several new instruments have been developed which enable the consumer to qualitatively and/or quantitatively evaluate the flavor and aroma barrier properties of food packaging films. Doyon et al. (26) evaluated a new automated, computerized instrument, the Aromatran, for the measurement of methyl ethyl ketone (MEK) at 23°C (26). The MOCON Aromatran was also performed to compare the flavor and moisture barrier of new barrier films with those of traditional coated films (27). Six films were ranked according to permeability and found that the barrier films performed better than traditional coextruded and coated papers.

SIGNIFICANT FINDINGS

When dealing with aroma compounds that interact with packaging material, the diffusion, solubility and permeability may have to be determined experimentally to enable prediction of the product's shelf life (27). Dow Chemicals, Co. has studied the diffusion of flavor, aroma, and solvent molecules through glassy polymers and olefin polymers (28). The best barrier properties were demonstrated by vinylidene chloride copolymers, polyethylene terephthalate, rigid polyvinyl chloride and polysulphone but not by olefin polymers. An equation was developed from Fick's First Law to illustrate the calculation of the permeability coefficient equivalent to the mass transport through a polymer at a steady state. It was shown that the diffusion of those molecules cannot be predicted simply from the rates of diffusion of oxygen through a particular polymer (28).

The procedures and materials used in a study on food packaging polymer films to evaluate the effect of relative humidity (RH%) on diffusion, permeation and sorption of the aroma compounds (aldehydes and alcohols), were also studied (29). The polymers investigated were linear low density polyethylene (LLDPE), high density polyethylene (HDPE) and ethylene vinyl alcohol copolymer (EVOH). Interactions between aroma and water vapor and the politicizing effects of water were the main cause of differences in diffusion, permeation and sorption. It was apparent that RH% did have a significant effect on the permeation and sorption properties in all the polymers. Diffusion of alcohols decreased, but in aldehydes diffusion increased when the humidity was high.

The barrier properties of food packaging materials are usually tested taking into account the effect of external parameters such as humidity, but not taking into consideration the effect of food parameters. Sorption of aroma compounds from food can affect food quality, flavor may be carried over in refillable plastic bottles, and sorption can also affect polymer properties and give the polymer new barrier characteristics. A specially designed permeation cell was used to make permeation measurements on aroma compounds presented as vapors (30). The aroma compounds were analyzed chromatographically. Humidity has an effect on the interaction between aroma vapors and polymers and the pH of the food affects sorption into the packaging material. Sorption of compounds varies when compounds are present together.

The effects of temperature, relative humidity and permeant concentration on the permeability of d-limonene in edible whey protein (WPI) polymer films were examined (30). Edible polymer films may be used as food coatings, film wraps or as supplements to synthetic wraps. Temperature and relative humidity demonstrated an exponential effect on d-limonene aroma transport in the films, although permeant concentrations between 62 and 226 ppm did not affect permeability.

Prediction methods for permeation through barrier materials based on Fick's Law, Henry's Law and Pasternak's equations were carefully studied (31). The dangers of predictions based on assumed Fickian behavior were also considered. Test methods have been developed that measure permeation, diffusion and solubility coefficients for different polymers and coatings. These methods can be applied to the assessment of polymer-permeant pairs, and thus the permeation of flavors and aromas through packaging materials. Tests must be carried out at precisely specified temperatures, humidities and permeant levels. The barrier properties of ethylene vinyl alcohol, for example, improved against printing solvent 2-butanone until a relative humidity of about 70% was reached, after which it began to degrade. Apparatus exists that will create a permeant, hold the correct environmental conditions, test the sample in duplicate, and display the results.

CONCLUSION

Unlike other permeation studies on inorganic materials, determining the permeability of organic vapors such as aroma is relatively complicated and the instrumental techniques are newly developed. In this paper, we introduced and discussed the importance of the study, theories and development of various instrumental technologies. Most studies were performed to evaluate new plastic materials and usually tested with food simulants such as toluene, limonene, menthol, and ethyl acetate. Theoretically, permeability of polymeric materials can be achieved by quantifying overall measurement of the diffusion and solubility, and by adopting Fick's law and Henry's law. However, it is more complex in actual field due to swelling behavior of organic vapors. Permeability of organic vapors through polymeric materials also increases exponentially by increase of temperature and relative humidity. Level of permeant and partial pressure are also important factors.

Because of the difficulty of permeation measurements of organic vapors, it is very difficult to get reproducible results in the actual experimentations. Trial-and-error process cannot be successful without setting a theoretical model of permeation about each organic vapor and polymeric material prior to the study. The researcher must also have good understanding of the nature of organic vapors and polymers too.

Permeability can be measured either by continuous methods (isostatic method) or accumulated methods (quasi-isostatic method). Nevertheless, permeability studies for organic vapors were developed dramatically from simple sensory evaluation to the edge of modern technologies such as gas chromatography and Aromatran. The demands on continuous permeability studies on organic vapors through polymeric materials strongly arises because of social attention on safety of food packaging.

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