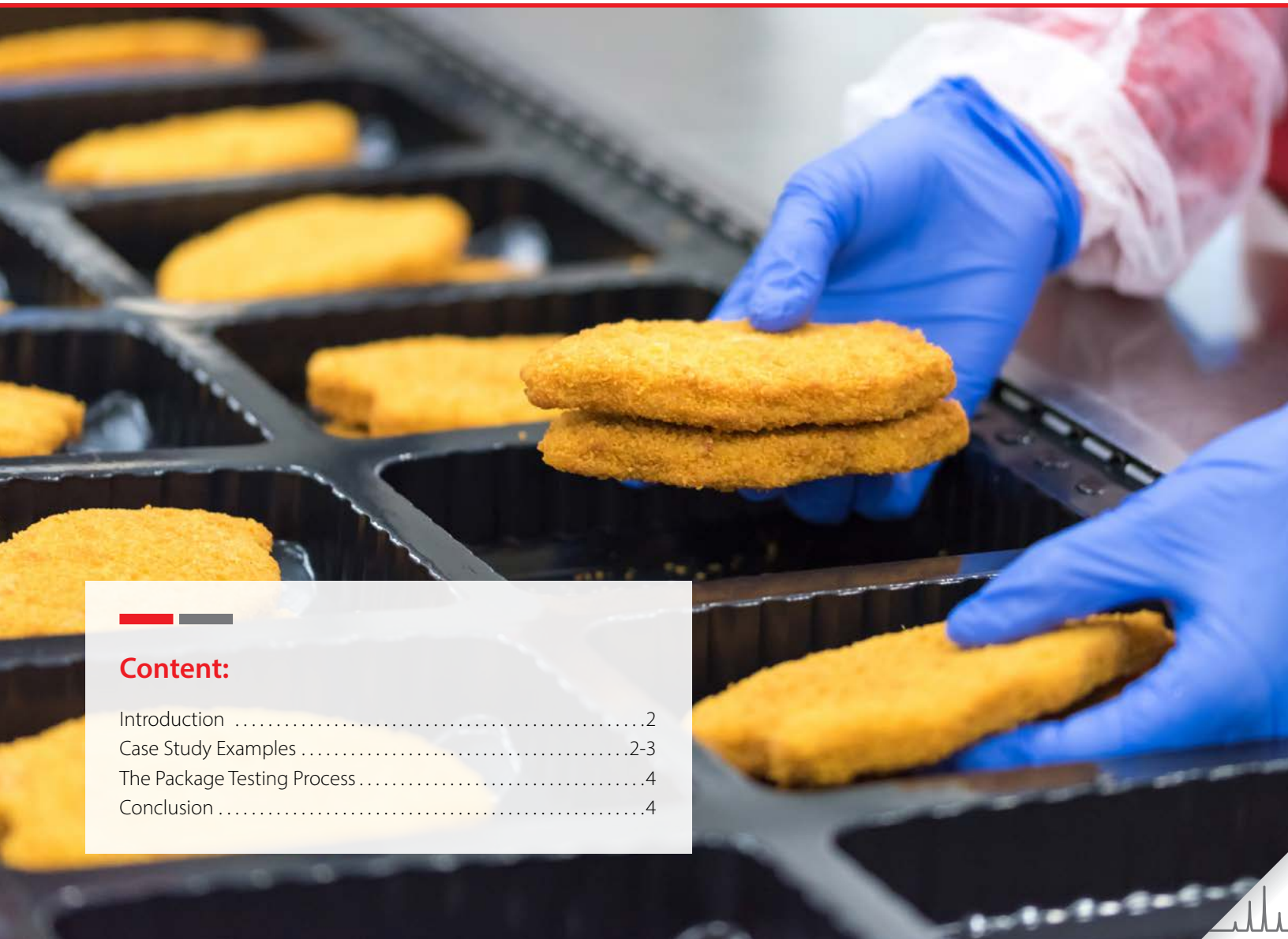
WHITE PAPER

SHELF LIFE AND THE IMPORTANCE OF TESTING THE WHOLE PACKAGE

AMETEK MOCON

August 2023



Content:

Introduction 2
Case Study Examples 2-3
The Package Testing Process 4
Conclusion 4

BARRIER DIFFERENCES: FLAT MATERIALS VS FINISHED PACKAGES

When a film is formed into a package, defects created during the manufacturing process and shipping or distribution can weaken the barrier provided by the package as a whole.

Introduction

Most packaging R&D work is done using flat films, which is essential for identifying suitable packaging materials. However, when a film is formed into a package, defects created during the manufacturing process as well as during shipping or distribution can weaken the barrier provided by the package as a whole. This must be taken into account during development or the shelf life of the product may be less than expected, leading to problems such as recalls or even legal action. Many companies avoid this issue by over-packaging their products. This leads to increased production costs, higher costs for the customer, and a negative environmental impact. A more effective solution is to determine the permeation rate of the finished package to ensure that it remains an effective barrier while avoiding the costs of over-packaging. The following case studies illustrate the importance of testing the whole package, and provide examples of how this type of testing can be done.

Case Study 1: Flat Film vs. Finished Package

An infant formula manufacturer wanted to switch from the traditional canister packaging to a multi-layer flexible pouch. Five candidate materials for these pouches are described in Table 1. Infant formula is moisture and oxygen-sensitive, so the water vapor transmission rate (WVTR) and oxygen transmission rate (OTR) into the pouches must both be determined. Since the destination market is in a tropical region, the test conditions for the films and packages were set as described in Table 2 using dry nitrogen as a carrier gas. All tests were performed on AMETEK MOCON® permeation testing instruments.

The film test results were all below the instruments' detection limits, indicating that they were effective barriers to both oxygen and water vapor. Using these film results and the actual size of the pouches, the pouches were predicted to have an OTR below 0.0004 cc/(pkg • day) and a WVTR below 0.0004 g/(pkg • day). However, once the completed pouches were tested, both the OTR and WVTR were significantly higher than anticipated (Table 3).

Examination of the pouches revealed that there were defects along the crease of the package sidewall, which allowed more water vapor and oxygen to permeate into the package than was expected (Fig. 1). It was only through testing completed pouches that the manufacturer was made aware of this issue.

Table 1. Candidate Materials

ID	Sample Structure
A	PE T/ A1 / Nylon / LLDPE
B	PET / A1 / PE
C	PE T/ A1 / PETMET / PE
D	PE T/ A1 / Nylon / PE
E	PE T/ PE (extruded) / A1 / PE (extruded) / PE

Table 2. Test Conditions and Equipment

Test Item	Test Conditions
Pouch OTR cc/(pkg•day)	Temperature: 37°C Test gas: 100% O_2 with 90%RH
Film OTR cc/(m^2•day)	Temperature: 37°C Test gas: 100% O_2 with 90%RH
Pouch WVTR g/(pkg•day)	Temperature: 37°C Test gas: 100% RH water vapor
Film WVTR g/(m^2•day)	Temperature: 37°C Test gas: 100% RH water vapor

Table 3. Pouch Test Results

Material ID	OTR cc/(pkg • day)	WVTR g/(pkg • day)
Projected	0.0004	0.0004
A	0.011	0.0021
B	0.032	0.0010
C	0.040	0.0022
D	0.036	0.0034
E	0.052	0.0015

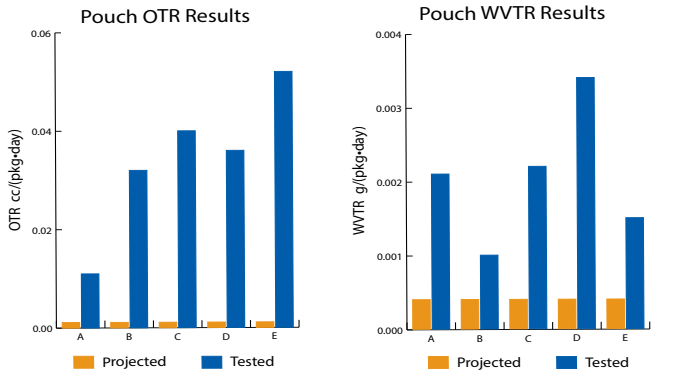


Figure 1. Projected vs. tested results

FINISHED PACKAGE BARRIERS: ONLY AS GOOD AS THE WEAKEST LINK

Case Study 2: Bottle vs. Closure

A health supplement is packaged in polymer bottles with simple cap closures. Permeation testing was conducted on bottles with and without these closures to determine how they impact the WVTR. To test bottles with closures, nitrogen gas lines were inserted into the bottle (Fig. 2). After the bottles were purged of oxygen, the tests were allowed to run to equilibrium. To test only the bottle body with no closure, the bottle was affixed to a metal plate using epoxy (Fig. 3), after which it was purged of oxygen and allowed to run to equilibrium.

Figure 2: Testing the bottle with closure

Figure 3: Testing the bottle body

Table 4: Bottle Test Results

Sample Name	WVTR g/(pkg • day)
W-1 (body and closure)	0.0070
W-2 (body and closure)	0.0089
W-3 (body and closure)	0.0099
W-4 (body and closure)	0.0042
W-5 (body and closure)	0.0107
W-6 (body only)	0.0030

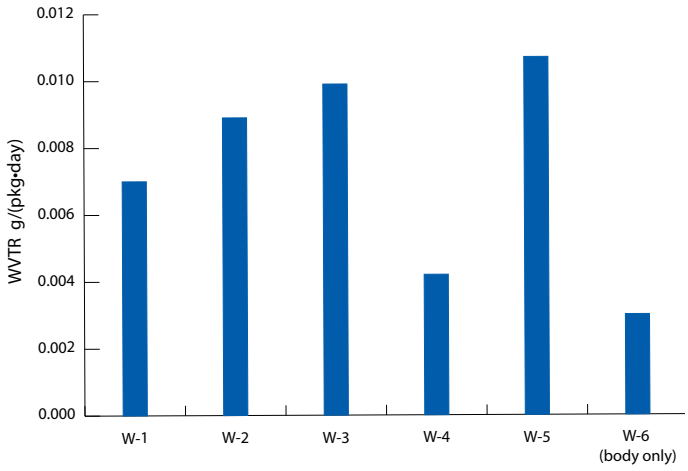


Figure 4. Bottle WVTR Test Results

Sample W-6 (without the closure) demonstrated the lowest WVTR, while that of bottles with the closure was much higher (Table 4 and Fig. 4). It is essential in the packaging development process to take this into account so that the supplement remains safe and effective when used by the customer.

Case Study 3: Retort Packaging, OTR Testing, and Shelf Life

Retort is the process of sterilizing a packaged food or beverage product in a modified pressure cooker containing hot water, steam, or a combination of both. During the retort process the oxygen barrier can change significantly through exposure to high heat and humidity (Fig. 5). The OTR of the samples was analyzed immediately after retort and remained in test until post-retort values were obtained.

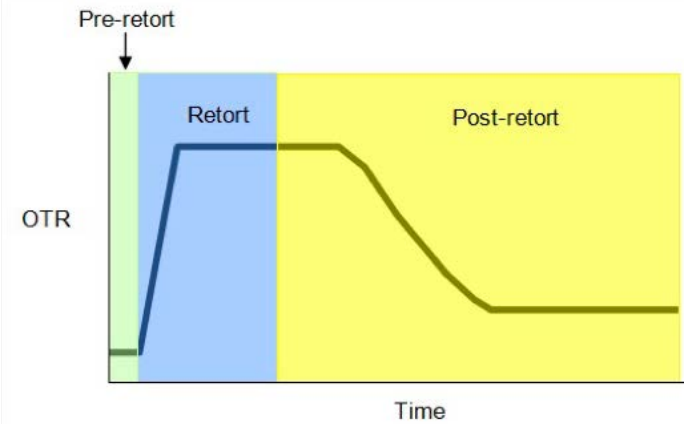


Figure 5. OTR pre- and post-retort

Figure 6 demonstrates the modeling of oxygen ingress over time by using the tested OTR value. A higher post-retort OTR can lead to a shorter shelf life for the product. For this reason, post-retort studies must be completed to determine the amount of oxygen entering the package following retort.

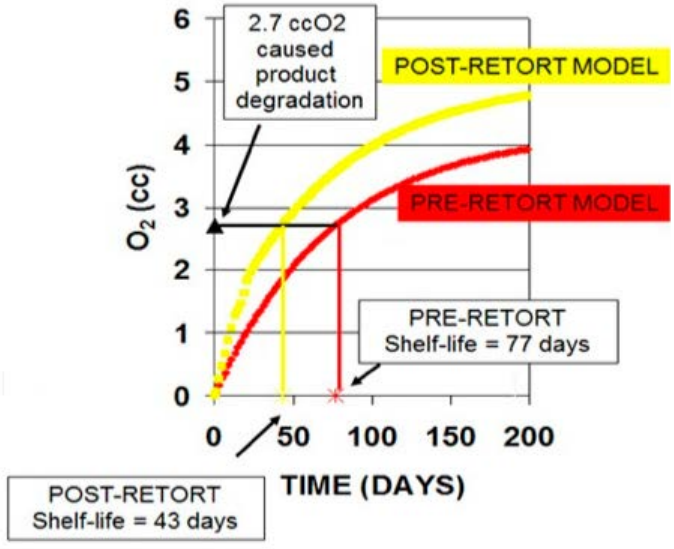


Figure 6. Shelf life prediction

THE IMPORTANCE OF WHOLE-PACKAGE PERMEATION TESTING

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The Package Testing Process

Testing packages operates on the same principles as testing films, but requires a special test setup. Figure 7 shows a common method for testing packages; while it shows a test measuring OTR, a similar test setup can be used for WVTR.

An empty package is sealed inside a bag or capture vessel and epoxied to a metal plate with gas lines going into the bag and package. The inside of the package is purged with N_2, and the test gas is then introduced into the bag or capture vessel surrounding the package. These traditional package testing options generally require a fair amount of preparation time and equipment. This has made them less popular, yet the results are crucial to ensuring your package will meet the needed performance.

To ease the burden of package sample preparation, AMETEK MOCON® offers a series of package testing cartridges that make mounting fast and easy. Some eliminate the need for epoxy entirely. Additionally, the simplified mounting methods allow for more repeatable testing results.

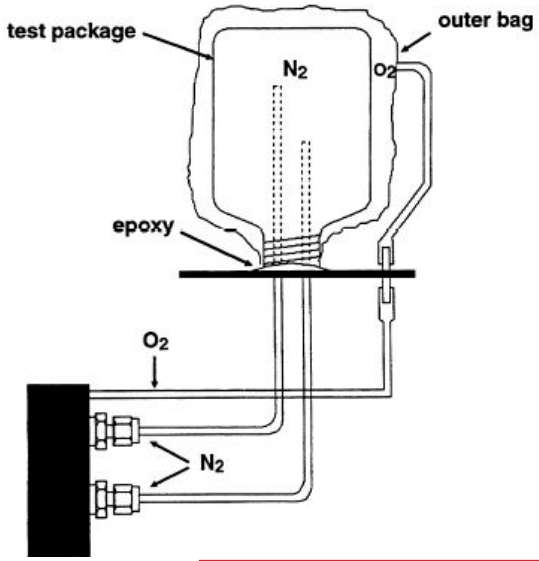

Figure 7. Package testing setup

Conclusion

Analyzing films to determine their OTR and WVTR is an essential part of the R&D process, but the actual permeation rates of finished packages can be much higher due to compromises and weak areas caused during manufacturing, shipping, and distribution. To understand the true permeation rate and resulting effect on shelf life of a package, it is essential to conduct permeation testing on the package as a whole.

AMETEK MOCON has the instruments and solutions to help make whole package testing easier and more reliable. As an example, Figure 8 shows the OX-TRAN 2/40 whole package permeation analyzer with a variety of different package testing cartridges loaded showing the versatility of these solutions.

Figure 8. The OX-TRAN 2/40 and AQUATRAN 3/40 enable testing of a variety of formed packages

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WP-PPS-3002-Shelf life and testing whole package-0923-1.2

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