Leak Detection in Polyethylene Terephthalate Bottles Filled with Water and Pulped and Unpulped Orange Juice Using a Vacuum Decay System

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ABSTRACT

This research evaluated an offline vacuum decay leak detection system for 1,775-ml polyethylene terephthalate (PET) bottles. These bottles were filled with water and pulped and unpulped orange juice and induction sealed with an aluminum liner and an outer 38-mm continuous thread polypropylene cap. The objectives of this study were to evaluate (i) minimum leak size sensitivity of the instrument; (ii) ability to identify weak but nonleaking seals; (iii) effect of varying fill heights on the equipment’s sensitivity; and (iv) percentage of false-positive and negative results likely to be obtained during a normal test run. To meet these objectives, leaks 5, 10, 15, 20, 30, 40, and 50 μm were created in the PET bottles. A second set of bottles was induction sealed at high voltage and 1, 1.5, 2, 2.5, 3, 3.5, and 4 s of dwell time. A third set of bottles with good seals was filled with differing headspace measurements of brimful, 1, 2, 3, 4, and 5 cm. After optimizing the equipment, leak tests on random sets of leaking and nonleaking bottles showed 0.0% false-positive and 0.0% negative identifications. Results showed 5-μm minimum leak size detection for bottles filled with all products. Optimum seal conditions were >2 but <3 s at high voltage. Product fill heights >2 to ≤3 cm did not affect the efficiency of the equipment. These results show that this vacuum decay system has potential for use in identifying leaks in PET bottles used for food packaging.

To meet the need for leak detection in packaged food and pharmaceutical products, many leak detection devices have been researched. Many of them are now commercially available and are used for online leak detection when the intent is to inspect each package produced and remove all defects. Irrespective of the technique used to inspect and remove defective packages from a production line, the success of the detection system depends on the skill of the operator, the nature of the package, the product type, and the sensitivity and accuracy of the equipment. These factors are also important considerations in deciding what type of leak detection system to use in a given operation. Other factors to be considered include the equipment cost, speed of operation, nature of the packaged product, simplicity of operation, and availability of after-sales service.

This study evaluated the use of an offline vacuum decay system for the detection of leaks in 1,775-ml polyethylene terephthalate (PET) bottles used for orange juice packaging. The general principle of operation for a vacuum (force) decay leak detection system can be divided into three major phases. This does not include the correct positioning of the test samples within the equipment prior to the commencement of the leak test and the sample removal at the end of the test. Once the sample is ready for testing, the area to be investigated for leaks is sealed by an airtight device within the leak tester. For vacuum decay systems, as was the case in this article, the first phase of the test process is the creation of a vacuum around the test package. Once this vacuum reaches a predetermined level, it forces the content of the package (including the headspace gas) to move toward the source of the vacuum. However, the content of the package is prevented from leaving the inside of the container as long as its closure mechanism maintains its integrity. To test the ability of the closure mechanism to prevent this from happening, the force created by the vacuum is maintained for a predetermined time period. This is the second part of the leak test and is referred to as a stabilization phase by some researchers (6). During this pressure holding period, it is not unusual for some of the vacuum (force) to decay (or be lost) from good packages. This will occur because the vacuum will dislodge air that is loosely attached to the outside of the package. In addition to this, some settling and creep will occur within the test package. If there are no leaks in the package and the closure mechanism is not disrupted, this loss of vacuum will be minimal. If the integrity of the closure is lost or if there are leaks in the package, a substantial amount of the vacuum will be lost. An evaluation of the amount of vacuum lost during a given test is the third part of the leak detection process. This third part is called the test phase. To determine if a package is defective, the amount of vacuum lost is compared with a predetermined threshold value. The package is defective if the vacuum lost exceeds this threshold value.

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The loss of vacuum during a leak test on a given package can be compared with the molecular flow of the headspace gas in a leaking container. This molecular flow through the leak is proportional to the difference in pressures applied across the leak. This can be expressed by use of Knudsen’s Law for molecular flow through a cylindrical tube. This law states

$$Q = 3.342 \left( \frac{r^3}{L} \right) \left( \frac{RT}{M} \right)^{1/2} \Delta P \tag{1}$$

where $Q$ is the leakage rate, $r$ is the radius, and $L$ is the length of the leak, respectively, and $M$ is the molecular weight of the gas, $T$ is the absolute temperature (°K), $R$ is the gas constant (8.315 J/mol in °K), and $\Delta P$ is the change in vacuum pressure (5). This rate loss of the content of the package during the stabilization stage of the test is also a factor of time. The longer the stabilization times during a normal leak test, the greater the loss of the contents and the better the chance of detecting the presence of a leak. This can be shown by

$$Q = \frac{\delta V}{\delta T} \tag{2}$$

where $\delta V$ is change in the contents of the package, and $\delta T$ is the time of the test.

The leak-susceptible part of the bottles studied in this article is the area of contact between the finish and the closure mechanism. In this case, the closure was an induction-sealed aluminum foil liner and a 38-mm continuous thread polypropylene (PP) outer cap. Leaks could also develop if the liner has pinholes, if the outer cap or the threads on the bottle finish are defective, or if the correct application torque is not applied when sealing the bottles. Various factors are known to influence the sensitivity and accuracy of vacuum decay leak detection systems. These include, but are not limited to, the viscosity of the product, temperature fluctuations, speed of the test, diameter and length of the defect, variability in headspace volume, and irregularities in the dimension of the package or the packaging materials. During this study, some of these issues were addressed in evaluating the vacuum decay leak detection tester. Thus, the selection of the product that filled the bottles included water and pulped and unpulped orange juice. This was done because of the potential for the pulp in the juice to plug small leaks. We sought to determine if the equipment was capable of detecting leaks with such difficult products.

Thus, the objectives of this study were to determine (i) the minimum size of leaks that can be detected by this device; (ii) the effect of changing headspace measurements on the sensitivity of the tester; (iii) the ability of the system to detect a nonleaking but weak seal; (iv) the potential of the tester to produce false-positive or -negative results; (v) the potential of the detector to damage good packages during a normal test; and (vi) the sensitivity of the detector for leak detection in bottles filled with products having different levels of particulates.

**MATERIALS AND METHODS**

The vacuum decay leak detector. The packaging integrity testing device used in this study was a Wilcomat R BS/V vacuum decay leak detector (U.S. patent 5,535,624 of 16 July 1996) manufactured by Wilco AG (Wohlen, Switzerland). This equipment, shown in Figure 1, is designed with a fixture head that can be lowered to cover the cap of a bottle to be tested. This head is fitted with a blow-up seal that creates a hermetically sealed testing area around the cap of the bottle once the test begins (see Fig. 2). This sealed area is then evacuated for a predefined time, and any vacuum change caused by a leak is monitored and registered. During the evacuation time, the actual vacuum curve is constantly monitored to detect the presence of large leaks. For a large leak, the test is immediately aborted by the software so that the content of the bottle is prevented from contaminating the equipment. If this bottle has a microleak, the extent of the vacuum decay is proportional to the size of the leak in the closure. For a leaking bottle, it would be identified as defective if the extent of the decay exceeded a preset value. Good nonleaking bottles, on the other hand, would be expected to show a vacuum decay value that does not exceed the preset value.

**Test package.** The packages tested in this study were 1,775-ml PET bottles filled with water, pulped orange juice, and unpulped
orange juice to a headspace depth of approximately 3 cm. The mouth of each test bottle was induction sealed with an aluminum liner and covered by a 38-mm continuous thread PP cap. The aluminum liner and the cap were applied to the bottle separately. A total of four types of samples were prepared. These sample types were nonleakers, liner leakers, samples filled with variable headspace volumes, and samples with weakly sealed liners and caps.

Sample preparation: nonleakers (good samples). To seal the test bottles, all liners were placed over the mouth of the container, and then the PP cap was screwed over the liner. All caps were tightened with 34-Nm torque with a SecurePak model 100 torque tester (Maumee, Ohio). This force was used because it was previously determined that it was sufficient to properly tighten the caps. The liners on the bottles were then sealed in place with a model ML0045 Auto Jr. Enercon induction sealer (Menomonee Falls, Wis.). To perform this, the bottles with the liner and cap were positioned under the sealer as described by the manufacturer. Once the sealer was activated, it sealed the liner to the mouth of the bottles while the cap is still in place. To obtain hermetic seals, the sealer was set at high voltage for a 2.5-s dwell time.

The 34-Nm torque value used as the optimum application force for tightening the cap on the bottles was determined after performing drop test analyses on samples filled with water, pulped orange juice, and unpulped orange juice. All bottles were filled with a 3-cm headspace measurement. These drop tests were similar to the ones done by Pascall (6) and Sivaramakrishna et al. (7). These researchers used modifications of the ASTM D-5276-00 and ASTM D-2463-90 methods with a model PDY-56 drop tester manufactured by Lansmont Corporation (Monterey, Calif.) (1, 2). To perform these tests, five sets of bottles were selected. These bottles were sealed with 6.8-, 13.6-, 20.4-, 27.2-, and 34-Nm torque and replicated three times. These packages were drop-tested according to the sequence described by the ASTM D-5276-00 method (2). Once the test was completed, each package was examined for signs of leaks by conductivity testing with a model KM-66 Kyoristu conductivity meter (Kyoritsu, Japan). All leaking bottles were further tested to confirm the result of leak testing by the dye test method. Dye testing allowed us to see the location of the leaks. The optimum torque selected was the one that produced sealed bottles with the least numbers of leaks. This torque or moment of the sealing force can be determined from the downward force that the cap applies to the mouth of the bottle multiplied by the perpendicular distance of the force (4). This can be expressed as

\[ T = Fr \]  
(3)

where \( T \) is the torque, \( F \) is the downward force, and \( r \) is the distance (3). However, the finish of the bottles complicates this distance \( r \) because it has to take into account the radii, threads, and frictional forces associated with the meshing of the PP cap to the bottle finish. Thus, for the bottle in our study, this application torque is given by the following formula:

\[ T_A = F_s \left[ \frac{r_i \cos \theta + 2\pi \mu_s r_i}{\cos 02\pi r_i - \mu \rho} \right] + \mu_s r_s \]  
(4)

where \( F_s \) is the vertical sealing force, \( r_i \) is the mean radius of thread contact, \( \theta \) is the thread-bearing angle, \( \rho \) is the thread pitch, \( r_i \) is the mean radius of the sealing surface, and \( \mu_s \) and \( \mu_r \) are the coefficients of friction at the thread interface and sealing surface. Assuming a theoretical system with zero friction and no time for stress relaxation of the liner, the vertical sealing force of the outer PP cap against the mouth of the bottle will be as follows:

\[ F_s = T_A \frac{2\pi}{\rho} \]  
(5)

This force will change, depending on the materials used in the closure and the weight of the bottle (8).

To determine the height from which the bottles should be dropped for this test, the mean failure height was established. Because this measurement was unknown at the start of the study, it was determined after several trials by the ASTM D-2463-90 and D-5276-00 methods described by Sivaramakrishna et al. (1, 2, 7). Once performed, this mean failure height was determined to be 15.24 cm.

The making of defective samples: liner leakers. To create these defects, a small hole was first made in the center of the liner. This hole was then covered with a stainless steel disc made with a laser-drilled hole (microleaks) of known size. All discs were fastened to the inside of the liner with a waterproof tape. The sizes of microleaks used for this test were 5, 10, 15, 20, 30, 40, and 50 µm. Each disc had only one hole and was obtained from Lenox Laser (Glen Am., Md.). All liners were hermetically sealed to the bottles. Prior to sealing the bottles, they were filled with water, pulped orange juice, or unpulped orange juice. Nine replicates of each leak size and each food type were prepared for this study.

Samples with weak seal without microleaks. These bottles were sealed with nonleaking aluminum liners. Although the liners were sealed with the outer caps tightened with 34-Nm torque, different dwell times were used. These were 1.0, 1.5, 2.0, 3.0, 3.5, and 4.0 s. Prior to sealing the bottles, they were filled with water, pulped orange juice, or unpulped orange juice. For each dwell time and food type, three samples were prepared. This test was replicated three times.

Optimization of the leak detection machine. The Wilcomat R BS/V leak detector was equipped with a flow meter and a needle valve that were used to calibrate and optimize the machine. The flow meter is calibrated to allow a known rate of air to enter the equipment. This rate of air was adjusted with the needle valve fitted to the equipment. To calibrate the leak detector with these built-in tools, a "dummy" bottle was placed in the sample holder normally used for testing containers. The needle valve used for this calibration was adjusted to simulate a 10-µm leak. The leak detector was then adjusted so that it could recognize this 10-µm leak. This was done by varying the rate of formation and the extent of the vacuum and stabilization time during the leak test. It was determined that a pressure of 5 × 10⁻⁴ Pa was required, and this produced a leak rate of 0.0181 cm³/s. Once this was finished, the dummy bottle was removed from the tester; and the needle valve was turned off. A 1,775-ml PET bottle fitted with a 10-µm leak and filled with water to a 3.0-cm headspace depth was then placed in the sample holder, and this container tested for the presence of the leak. The leak detector was fully optimized after it showed the ability to detect the 10-µm leak in this test sample. A time of 18 s was required to test each bottle.

To confirm that the equipment was optimized for leak detection in the 1,775-ml PET bottles filled with water to a 3.0-cm headspace depth, 300 defect-free bottles were tested. These were filled with water, pulped orange juice, and unpulped orange juice. These comprised 100 bottles filled with each food type. In addition to these, 12 bottles with each food type were prepared, and each bottle was fitted with three 20-, 30-, 40-, and 50-µm liner leaks. These were then randomized with the nonleaking bottles, and all samples were tested on the leak detector. The main objec-
tive of this was to determine the ability of the detector to identify leaks in randomly arranged samples.

Variable fill height (headspace) measurements. This test was done by preparing PET bottles filled with water, pulped orange juice, and unpulped orange juice. For each food type, the headspace depth measurements were 0, 1, 2, 3, and 4 cm. All bottles were closed at the optimum sealing and capped conditions. These optimum conditions ensured that the samples had a low potential to develop leaks. For each food type and each fill height measurement, the samples were prepared in triplicate. All tests were repeated three times. These samples were tested on the leak detector set at its optimum sensitivity, and the vacuum loss values were recorded. For all samples identified as defective by the leak tester, a conductivity leak test was performed to ensure that no leaks developed in the samples during the test.

The potential of the leak detector to damage good PET bottles during a normal test. This evaluation was done by preparing 150 defect-free PET bottles filled with water, pulped orange juice, and unpulped orange juice. Each food type had 50 bottles. All bottles were sealed at the optimum conditions for the aluminum liner and the torque for the outer cap. The use of the optimum sealing conditions was necessary to minimize any chances of defective seal formation and subsequent failure of the seals during the test. All bottles were leak tested on the vacuum decay leak detector. The intent of this test was to investigate the potential of the detector to weaken good seals during a normal test. In addition to this, the leak tester was adjusted to its optimum sensitivity for leak testing of the PET bottles. After leak testing, all samples were manually conductivity leak tested to determine if the leak detector caused any leaks to develop in the seals. If the conductivity test showed samples with leaking seals, a manual dye test was done to determine the exact position of the leak.

Conductivity test for PET bottles. To perform this test, a sample bottle was cut into two halves. One half had the bottle finish together with the closure mechanism. The other half had the bottom of the bottle and was discarded. The part of the bottles (with the closure) was then air dried to prevent the creation of false-positive or negative results. This was then conductivity tested in a manner similar to the method reported by Pascall (6). This conductivity test is sensitive to leaks in bottles that are as small as 1 μm in diameter.

Dye penetration testing of PET bottles. The dye penetration leak test is done to show the presence and location of a leak. The dye used in this study was a low-surface-tension red organic solvent obtained from Magnaflux Inc. (Troy, Mich.). Prior to commencing the test, the sealing area of each sample was air dried. About two to three drops of the penetrant dye were then placed around the seal and on the aluminum liner, making sure that the entire closure was exposed to the dye. After 2 h of drying and allowing the dye to penetrate all areas of the closure, the outer cap was removed, and a visual inspection was done to see if any colored pathways existed. This would indicate that the bottle was leaking. The dye test is capable of detecting leaks as small as 5 μm in diameter.

Detection of false-positive and false-negative results produced by the leak tester. This test was done in conjunction with the optimization routine of the equipment. During the optimization, 300 defect-free bottles were prepared and leak tested on the leak tester. For the false-positive and -negative tests, these bottles were conductivity tested to see if they showed up as leaking. This indicated a false negative if they did leak during the conductivity test. For the leaking samples prepared for the optimization, any bottles that were not detected as leaking would be false positive.

Detection of weak but nonleaking seals in test packages. This test was done by filling PET bottles with water, pulped orange juice, and unpulped orange juice. The bottles were then sealed with the aluminum liner and the PP outer cap. The application torque on the cap of all the samples was 34 Nm; the sealer was set at high voltage, but the dwell times varied at 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, and 4.0 s. Three samples were prepared for each dwell time and for each food type. All samples were leak tested on the vacuum decay leak tester. This test was repeated three times.

Statistical analysis. The ability of the leak detector to identify a leak of a certain size in the PET bottles was determined by dividing the number of successful detections by the total number of packages tested. This was done for each leak size and for each packaged food type. In addition to this, calculations were done to estimate the uncertainty associated with the detection rate for each leak size and the associated food types. To perform this estimate, the data were first assumed to be binomial for each test. This meant that each defective package had some fixed probability of being correctly detected. The method of calculating the probability, variance, and uncertainty associated with the detection rate was similar to that used by Sivaramakrishna et al. (7).

RESULTS AND DISCUSSION

The optimum sealing condition for the PET bottles. The optimum sealing conditions for the water and pulped and unpulped orange juice bottles are shown in Figure 3. This figure shows that the high voltage settings for 2.5 s provided the bottles with the best seal quality. The optimum torque for a sufficiently tight cap was determined to be 34 Nm. This was obtained from drop test studies performed during a previous study on the 1,775-ml PET bottles (7).

Data showing the vacuum decay values for the water, unpulped orange juice, and pulped orange juice bottles are shown in Figures 4, 5, and 6, respectively. This represents the sensor values for these samples filled at the optimum headspace measurement of 3 cm and sealed with 34 Nm torque for 2.5 s. All samples used for this optimization test were leak-free 1,775-ml PET bottles. This was determined by conductivity and dye testing after leak testing the samples with the vacuum decay tester. For all samples tested

FIGURE 3. Vacuum decay sensor readings for PET bottles sealed at varying conditions.

\[ \text{Vacuum decay readings for PET bottles.} \]

145 vacuum decay

<table>
<thead>
<tr>
<th>Voltage</th>
<th>Voltage</th>
<th>Voltage</th>
<th>Voltage</th>
<th>Voltage</th>
</tr>
</thead>
<tbody>
<tr>
<td>water</td>
<td>OJ no pulp</td>
<td>OJ pulp</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.0 sec high</td>
<td>2.5 sec high</td>
<td>3.0 sec high</td>
<td>3.5 sec high</td>
<td>4.0 sec high</td>
</tr>
</tbody>
</table>

Sealing conditions

<table>
<thead>
<tr>
<th>Voltage</th>
</tr>
</thead>
<tbody>
<tr>
<td>water</td>
</tr>
<tr>
<td>OJ no pulp</td>
</tr>
<tr>
<td>OJ pulp</td>
</tr>
</tbody>
</table>

Statistical analysis.

The ability of the leak detector to identify a leak of a certain size in the PET bottles was determined by dividing the number of successful detections by the total number of packages tested. This was done for each leak size and for each packaged food type. In addition to this, calculations were done to estimate the uncertainty associated with the detection rate for each leak size and the associated food types. To perform this estimate, the data were first assumed to be binomial for each test. This meant that each defective package had some fixed probability of being correctly detected. The method of calculating the probability, variance, and uncertainty associated with the detection rate was similar to that used by Sivaramakrishna et al. (7).
in this part of the study, none produced vacuum decay values greater than 145 U. As a result, the 145 vacuum decay value was chosen as the maximum threshold for nonleaking samples. Any test sample producing a decay value above 145 U was then regarded as defective. This optimum sealing condition was also confirmed by the frosty appearance of the sealing surface when the aluminum liner was pulled away to access the contents of the bottles. This is what is normally done by a consumer who wants to consume the contents of the bottle. This frosty appearance represents the melting and fusing of the plastic layers on the liner and the mouth of the bottle. The intensity of the frostiness was largest at the optimum sealing condition, and this was used as the initial estimate for a good seal. This was later confirmed by testing and will be explained in a later section.

**Leak sizes identified by the leak detector.** All leak tests performed on the bottles were done on samples sealed with the aluminum foil liner. The leak sizes created in these liners were 5, 10, 15, 20, 30, 40, and 50 μm. Table 1 shows the results for the different leak sizes that were detected. All bottles were sealed at the optimum condition, and this allowed us to conclude that no leaks occurred from poorly sealed surfaces. The results in Table 1 show that all leaks ≥5 μm were detected in the water-filled bottles. For the pulped orange juice, none of the 5-μm leaks was detected, 78% of the 10-μm leaks were detected, and all leaks ≥15 μm were detected by the tester. For the unpulped orange juice, 72% of the ≥5-μm leaks were detected, and all leaks ≥10 μm were detected.

These leak size results show that the greater the amount of particulates in a liquid used to fill a bottle, the more difficult it would be to detect microleaks in that container. This can be concluded by the observation that all leaks in bottles filled with water were detected. However, 72% of the 5-μm leaks were detected when the water was replaced with orange juice without pulp. For the orange juice with pulp, we see that none of the 5-μm leaks were detected and that only 78% of the 10-μm leaks were identified by the
Vacuum decay sensor readings for PET bottles filled with pulped orange juice.

FIGURE 6. Vacuum decay sensor readings for PET bottles filled with pulped orange juice.

These results were corroborated by the probability and uncertainty calculations, which show that the packages filled with water had a higher rate of detection for leaks when compared with the orange juice–filled bottles. The detection rate for the unpulped orange juice was higher than for the pulped juice.

These results are also consistent with the Poiseuille’s equation. This equation describes the steady (laminar) flow of a liquid through a given leak. The Poiseuille equation shows that the flow through the leak decreases with an increase in the viscosity of the liquid, assuming that all other conditions remain constant. This equation is expressed as

\[ Q = \frac{\pi \Delta p a^4}{8\eta L} \]  

where \( Q \) is the volumetric rate of flow (in cubic meters per second), \( \Delta p \) is the pressure difference, \( L \) is the length of the leak, \( a \) is the radius, and \( \eta \) is the viscosity of the liquid within the package. In addition to the viscosity, the fibrous pulp in the orange juice may have acted to plug the smaller size leaks and thus help produce the results shown in Table 1.

\[ \text{TABLE 1. Percent leak size detected by the vacuum decay tester for PET bottles}^a \]

<table>
<thead>
<tr>
<th>Leak size (( \mu m ))</th>
<th>% detected as defective</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PET bottles (water)</td>
</tr>
<tr>
<td>5</td>
<td>100</td>
</tr>
<tr>
<td>10</td>
<td>100</td>
</tr>
<tr>
<td>15</td>
<td>100</td>
</tr>
<tr>
<td>20</td>
<td>100</td>
</tr>
<tr>
<td>30</td>
<td>100</td>
</tr>
<tr>
<td>40</td>
<td>100</td>
</tr>
<tr>
<td>50</td>
<td>100</td>
</tr>
</tbody>
</table>

\[ ^a \text{OJ, orange juice.} \]

\[ \text{TABLE 2. Percentages of PET bottles rejected as defective on the basis of sealing conditions}^a \]

<table>
<thead>
<tr>
<th>Sealing (s) (high voltage)</th>
<th>% detected as defective</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PET bottles (water)</td>
</tr>
<tr>
<td>1.0</td>
<td>100</td>
</tr>
<tr>
<td>1.5</td>
<td>100</td>
</tr>
<tr>
<td>2.0</td>
<td>100</td>
</tr>
<tr>
<td>2.5</td>
<td>100</td>
</tr>
<tr>
<td>3.0</td>
<td>100</td>
</tr>
<tr>
<td>3.5</td>
<td>100</td>
</tr>
<tr>
<td>4.0</td>
<td>100</td>
</tr>
</tbody>
</table>

\[ ^a \text{OJ, orange juice.} \]

Determination of false-negative and false-positive results. The dye test showed that all leaks occurred through the artificially created leaks. In addition to this, all samples that were not designated as leaking by the leak detector were conductivity tested. This test showed that all samples identified as leaking by the vacuum decay leak detector also showed positive by the conductivity test. The result of this second conductivity test also showed no signs of leaks in these bottles. Thus, we concluded that the vacuum decay leak detector produced no false-negative or false-positive results when leaks \( \geq 20 \mu m \) were used to challenge the equipment.

Determination of the ability of the system to detect a nonleaking but weak seal. This test was done to identify seals that had no pinhole microleaks but that may have been seals with suboptimum conditions of temperature, dwell time, or pressure. Optimization of these conditions is essential for proper induction sealing of the aluminum liner to the mouth of the bottles. The heat produced by the voltage setting of the sealer, the pressure applied to the sealing surfaces by the application torque of the PP cap, and the time of sealing all work together to produce a proper seal. Any attempt to seal the bottles under less than ideal sealing conditions
conditions can result in weak or burnt seals, even though there may not be any pinholes in the liner. Thus, these seals may not show signs of leak at the time of fabrication but can be susceptible to failure when exposed to the stresses of transportation and handling during retail trade.

Results showing the ability of the leak detector to identify bottles with weak but nonleaking seals are shown in Table 2. These results are fairly similar for all samples. It showed that bottles sealed for 3.0 s showed no signs of failure. For the samples sealed at 2.5 s, a little more than half showed signs of failure. All samples sealed ≤2.0 s and ≥3.5 s showed signs of failure during the leak test. It is obvious from these results that the samples sealed for ≥2.0 s failed because there was insufficient time for the bonding of the polymer on the liners and the mouth of the bottles. When the liners on these bottles were pulled apart, they came off very easily and with little resistance. For the samples sealed for ≥3.5 s, failure of the seal occurred because exposure to the sealing heat was too long, which resulted in burning of the polymer. This was quite evident during a visual inspection of these seals.

Effect of varying fill height on the sensitivity of the online leak detector. Results for this test are shown in Table 3. The results show that PET bottles filled to a depth of 3 cm were not identified as defective. Figure 7 shows the vacuum decay values for each fill height. Bottles with headspace measurements below or above this depth were categorized as defective by the leak detector. This also included all bottles that were filled without headspace (brimful). This result showed that the leak detector was also capable of identifying bottles that were incorrectly filled. The equipment could also be used to establish the optimum fill height for the test bottles. Thus, the use of this equipment can eliminate the need for a separate fill height monitor.

From the results obtained in this study, the following conclusions can be drawn. (i) For PET bottles filled with water, the minimum leak size for 100% detection was 5 μm. (ii) For PET bottles filled with pulped orange juice, the minimum leak size for 100% detection was 15 μm. (iii) For PET bottles filled with filtered orange juice, the minimum leak size for 100% detection was 10 μm. (iii) The vacuum decay detector determined the optimum sealing conditions to be 2 to 3 s at the induction sealer setting of high voltage. (iv) The vacuum decay detector determined the fill height measurement of the 1,775-ml PET bottles for optimum leak detection to be 3 cm. (v) There were no false-positive or false-negative results when the PET bottles were tested for leaks <15 μm. (vi) The leak size detection sensitivity of the tester was inversely proportional to the degree of particulates in the products used to fill the bottles.

### Table 3. Percentages of nonleaking rejected bottles filled at varying height measurements

<table>
<thead>
<tr>
<th>Fill height (cm)</th>
<th>PET bottles (water) % detected as defective</th>
<th>PET bottles (OJ no pulp) % detected as defective</th>
<th>PET bottles (OJ pulp) % detected as defective</th>
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<td>Brimful</td>
<td>100</td>
<td>100</td>
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</tr>
<tr>
<td>1.0</td>
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<td>100</td>
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</tr>
<tr>
<td>2.0</td>
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<tr>
<td>3.0</td>
<td>0</td>
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</tr>
<tr>
<td>4.0</td>
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<td>56</td>
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</tr>
</tbody>
</table>

*a* OJ, orange juice.

### References


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