What is the Best Way to Process Clear Retort Pouches Through the Value Chain?
Examining the Effects of Converting, Retorting and Drop Tests on Stand-up Pouches Containing SiOx-coated PET

Dante Ferrari
Celplast Metallized Products Ltd.

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Introduction

The retort pouch market has been an area of intense development over the past few years, as consumer goods companies are looking to provide more convenient, better tasting food to the consumer. Clear retort pouches have been making in-roads into applications where the ability to microwave, visibility of the product and metal detection capabilities are all important. In particular, oxide-coated films offer several benefits, such as no yellowing, no retort shock, full FDA approval and recyclability compared to other clear barrier films.

In spite of these advantages, one reason oxide-coated clear retort pouches have not gained increased market share is due to their cost. However, recent studies have shown that a 3-ply lamination can actually provide sufficient barrier properties after pouch-making and retorting without significantly increasing costs. This study goes a step further, to examine the effects on barrier properties of creating a stand-up pouch, putting it through different retort conditions, and looking at the effect of dropping it.

Experimental & Results

We began by working with a commercial converter to produce a printed 3-ply solvent-based laminated structure (48 g PET / 48 g SiOx-coated CERAMIS® PET / 3 mil CPP):

<table>
<thead>
<tr>
<th>Polyester</th>
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</thead>
<tbody>
<tr>
<td>Ink</td>
<td></td>
</tr>
<tr>
<td>Adhesive</td>
<td></td>
</tr>
<tr>
<td>Oxide-coated PET</td>
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</tr>
<tr>
<td>Adhesive</td>
<td></td>
</tr>
<tr>
<td>Cast Polypropylene</td>
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</table>
The commercial converter also prepared stand-up pouches with a flat-bottom gusset for evaluation. Barrier properties of the SiOx-coated PET were not compromised during adhesive laminating to form the rollstock, maintaining a value of 0.3 cc/m²/day. However, during stand-up pouch forming the barrier properties of the three faces of the pouch (front, back and bottom gusset) were all somewhat compromised:

- Front panel OTR = 1.3 cc/m²/day
- Back panel OTR = 1.1 cc/m²/day
- Bottom gusset OTR = 3.8 cc/m²/day

We then carried out a design of experiments (DOE) using a commercial-sized retort chamber. The steam conditioning and cooling times were standardized. The following variables were investigated in the DOE:

- Retort temperature – 121°C vs. 135°C
- Retort time – 15 min vs. 45 min
- Pouch liquid – water vs. bentonite slurry (convective vs. conductive heat transfer)
- Drop test – dropped vs. not dropped (ASTM D775.61.B)

Oxygen barrier testing was completed for all samples on both the front and back face. The raw data from the DOE was then compiled.

<table>
<thead>
<tr>
<th>Pouch Liquid</th>
<th>Retort Temp (°C)</th>
<th>Drop</th>
<th>Retort Time (min)</th>
<th>Back/Front Panel</th>
<th>OTR (cc/m²/d)</th>
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<tbody>
<tr>
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<td>135</td>
<td>N</td>
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<td>F</td>
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<tr>
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<td>N</td>
<td>45</td>
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<td>1.84</td>
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</tbody>
</table>
Initially, we added the front vs. back faces of the retort pouches as a fifth DOE variable. This led to several main factors and interactions being significant. This stemmed from the fact that at extreme retort conditions (high temperature, long retort time), the back panel barrier properties deteriorated significantly, while the front panel barrier properties did not. See the figure on the following page for a visual display of these interactions. We concluded that the overpressure acting on the back of the pouch against the support tray was leading to a distortion of the film, which created the compromised barrier.

We then re-evaluated the DOE using our original four variables under investigation. In this case, we found two primary effects on oxygen barrier: the top one was retort temperature, the lesser one was retort time.
It is interesting to note that neither dropping the pouches nor the heat transfer mechanism inside the pouch had a significant impact on the test results.

Conclusions

The adhesive lamination process did not compromise barrier properties of the original SiOx-coated PET film whatsoever. The stand-up pouch forming process did impact barrier properties, from three- to ten-fold, depending on whether we measured the barrier properties in the gusset or faces of the pouch.

The retort and drop test DOE showed that both retort time and temperature can have a significant impact on barrier properties. In particular, the physical constraining of a pouch in an overpressure situation should be minimized as much as possible, in order to retain acceptable barrier properties.

Acknowledgments

I would like to thank my colleague Steve Skopitz for carrying out the majority of testing and preparing the DOE reports. Also Allan Mannen of the Guelph Food Technology Center for use of their retort chamber, and Tom Mueller of Rohm & Haas and Scott Whiteside of Clemson University for their guidance.

References

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Outline

• Retort pouch market
• Clear retort pouch structure selection
• Clear retort trials - DOE
• Clear retort trials - barrier test results
• Conclusions
**Retort Pouch Market**

Drivers to replace cans & glass jars:
- Lighter weight
- Safer - no sharp edges
- Faster heating – leads to more even cooking & better taste
- 8 billion pouches in 2005, 15% annual growth rate

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**Retort Pouch Market**

Typical 4-ply MRE foil retort pouch structure:

<table>
<thead>
<tr>
<th>Layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyester</td>
</tr>
<tr>
<td>Polyethylene</td>
</tr>
<tr>
<td>Adhesive</td>
</tr>
<tr>
<td>Nylon</td>
</tr>
<tr>
<td>Adhesive</td>
</tr>
<tr>
<td>Aluminum Foil</td>
</tr>
<tr>
<td>Adhesive</td>
</tr>
<tr>
<td>Cast Polypropylene</td>
</tr>
</tbody>
</table>

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**Retort Pouch Market**

Drivers to use clear retort pouches:
- Visibility of product
- Microwavable
- Use with metal detectors
- Use with RFID tags
Retort Pouch Market

Drivers to use oxide-coated clear retort films:
• No yellowing
• Environmentally acceptable
• No “retort shock”
• Excellent chemical & odor resistance

Outline

• Retort pouch market
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Clear Retort Pouch Structures

Typical 4-ply clear barrier retort pouch structure:

- Polyester
- Adhesive
- Oxide-coated Polyester
- Adhesive
- Nylon
- Adhesive
- Cast Polypropylene
Previous Test Results – Rohm & Haas

4-Ply Clear Barrier Oxygen Transmission Rate Results

Clear Retort Pouch Structures

Why else use silicon-oxide coated PET?

• Good processability – in a dozen separate converting trials carried out in North America over past 18 months, SiOx-coated PET film barrier properties held up after printing and/or laminating

• Excellent flex-crack resistance, as measured using Gelbo flex tests, before and after retorting

• Approved under FDA regulation 21.CFR.§ 177.1390 for retort conditions up to 135°C

Typical 3-ply clear barrier retort pouch structure:

- Oxide-coated Polyester
- Adhesive
- Nylon
- Adhesive
- Cast Polypropylene
Clear Retort Pouch Structures
Possible 3-ply clear barrier retort pouch structures:

- Polyester
- Ink
- Adhesive
- Oxide-coated PET
- Adhesive
- Cast Polypropylene

Outline

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Retort Trials - DOE
Commercial converter prepared the following solvent-based lamination:
- reverse printed 48 g PET
- SiOx-coated 48 g PET
- 3 mil CPP sealant

Oxygen barrier of SiOx-coated PET was retained during laminating:
- Laminated sample OTR = 0.3 cc/m²/day
Retort Trials - DOE

- Converter also prepared stand-up pouches on Totani pouch machine:
  - 150 mm x 155 mm front & back panels
  - Flat-bottom gusset
  - Hold 300 g of product

- Oxygen barrier was partially compromised during pouch-making:
  - Front panel OTR = 1.3 cc/m²/day
  - Back panel OTR = 1.1 cc/m²/day
  - Bottom gusset OTR = 3.8 cc/m²/day

By: Dante Ferrari

Retort Trials - DOE

The Retort Chamber - Closed

By: Dante Ferrari

Retort Trials - DOE

The Retort Chamber - Open

By: Dante Ferrari
Retort Trials - DOE

• Retort cycle:
  • Introduce steam, vent air: 4 minutes
  • Circulate steam to increase temperature and pressure to set point: 3 minutes
  • Retort at steady state: 15 or 45 minutes
  • Introduce cooled steam: 3 minutes
  • Water spray: 40 minutes

• Controlled constants:
  • Same stand-up pouches & seals
  • Same liquid quantities (300 g)
  • Same tray configuration for pouches: always faced front of pouch upwards, bottom gusset towards retort chamber door
  • Maintained even pressure across pouches by using 25 psig at 121°C, 40 psig at 135°C

• Controlled variables:
  • Retort temperature – 121°C vs. 135°C
  • Retort time – 15 min vs. 45 min
  • Pouch liquid – water vs. bentonite slurry (convective vs. conductive heat transfer)
  • Drop test – dropped vs. not dropped (ASTM D775.61.B)
Retort Trials - DOE

$2^{4-1}$ Factorial Design
(screening experiment)

<table>
<thead>
<tr>
<th>Pouch Liquid</th>
<th>Temp ($^\circ$C)</th>
<th>Drop</th>
<th>Retort Time (min)</th>
</tr>
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<tbody>
<tr>
<td>W</td>
<td>130</td>
<td>N</td>
<td>15</td>
</tr>
<tr>
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<td>Y</td>
<td>15</td>
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<tr>
<td>B</td>
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<td>B</td>
<td>135</td>
<td>Y</td>
<td>46</td>
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<td>N</td>
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<td>W</td>
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<td>Y</td>
<td>45</td>
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<tr>
<td>W</td>
<td>121</td>
<td>N</td>
<td>45</td>
</tr>
</tbody>
</table>

Outline

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Retort Trials - Barrier Results

Raw Data

<table>
<thead>
<tr>
<th>Pouch Liquid</th>
<th>Retort Temp ($^\circ$C)</th>
<th>Drop</th>
<th>Retort Time (min)</th>
<th>Raw Data</th>
<th>OTR (in ml/mb)</th>
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</thead>
<tbody>
<tr>
<td>B</td>
<td>130</td>
<td>N</td>
<td>15</td>
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<td>W</td>
<td>121</td>
<td>N</td>
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</tbody>
</table>
Retort Trials – Barrier Results

• Since we had both the front and back panel face data at every condition, we could add the face as a fifth variable
• Factorial design temporarily became $2^5 - 1$, looking at 5 factors

Retort Trials – Barrier Results
Which factors were significant? Most of them!

Panel face interactions with retort time and temperature
Retort Trials – Barrier Results

• At high retort temperature and time, due to higher overpressure and the tray geometry, the back panel barrier properties were compromised

• We decided to change factorial design back to $2^{4-1}$, looking at the original four factors

Retort Trials – Barrier Results

Now which factors are significant?

Only retort temperature & time

<table>
<thead>
<tr>
<th>Factor Name</th>
<th>Standardized Effect</th>
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</thead>
<tbody>
<tr>
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<tr>
<td>D</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td></td>
</tr>
</tbody>
</table>

Pareto Chart of the Standardized Effects

(response is O₂ perm, Alpha = .05)

Retort Trials – Barrier Results

Other key findings:

• Drop test close to being significant, but did not create a large effect

• Interactions between all variables examined were definitely insignificant

• Experimental error based on repeats: standard deviation was 0.30 cc/m²/day
Conclusions

• SiOx-coated PET has retains its barrier properties when producing adhesive laminated rollstock
• During stand-up pouch forming, barrier properties can be compromised (3 - 10x increase in OTR)
• Retort conditions can play a role in further compromising barrier properties, particularly at high temperature and time

Conclusions

• Drop testing and mode of heat transfer within the pouch did not have a significant impact on barrier
• Loss of barrier in back panel of pouch after extreme time and temperature retorting: suggests minimizing physical pouch constraint in the retort tray and minimizing overpressure are critical to successfully retaining barrier after retort

Acknowledgments

• Allen Mannen of Guelph Food Technology Centre
• Steve Skopitz of Celplast Metallized Products
• Scott Whiteside of Clemson University