Improve the performance of your retort flexible film

FLEXPACKCON 2018
Phoenix, October 31st 2018

Sergi Salvà Sàez
Technical Development Manager
UBE America Inc.
Definition:
Retort is a **sterilization** process in autoclave under **high temperature and pressure**.
This process is also used in the food industry to **extend** the **shelf life** of goods.

\[
T \geq 121^\circ C \\
t \geq 30 \text{ min}
\]
Retort food

Ready to eat meals

Fish

Vegetables

Pet food
Requirements for retort packaging:

- Resist treatment temperatures and humidity
- Do not undergo undesirable changes (color, appearance, transparency)
- Heat-resistant seal system during heat treatment
- Do not have migrations
- Facilitate thermal transfer
- Barrier properties appropriate to the estimated Shelf-life of the food
Retort packaging

Agenda

- Retort – Process & Food
- O₂ Barrier vs moisture
- New retort packaging concept
- Evaluation methodology & Data
- Which UBE NYLON for retort?
- Case study
- Conclusions
High moisture content of the barrier layer = Lower Oxygen barrier
Moisture absorption in autoclave

In autoclave during retort process

- $T \geq 121^\circ C$
- $P$ : High
- $t \geq 30$ min

On the shelf after retort process

- $T$ = Room temp.
- $P$ : atm.
- $t$ : long storage

Moisture absorption of the barrier layers

Slow moisture release (drying)
Retort packaging

Agenda

- Retort – Process & Food
- O₂ Barrier vs moisture
- New retort packaging concept
- Evaluation methodology & Data
- Which UBE NYLON for retort?
- Case study
- Conclusions
Why not placing the barrier materials in outer layer?
In autoclave during retort process

- $T \geq 121^\circ C$
- $P : \text{High}$
- $t \geq 30 \text{ min}$

Moisture absorption of the barrier layers

On the shelf after retort process

- $T = \text{Room temp.}$
- $P : \text{atm.}$
- $t : \text{long storage}$

Slow moisture release (drying)

Moisture barrier of polymers
Moisture barrier of polymers

WVTR @ 40ºC, RH 90%

<table>
<thead>
<tr>
<th>WVTR (g/100in².day)</th>
<th>PP, 0.7</th>
<th>NYLON, 11</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>PP</td>
<td>NYLON</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>12</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Test according JIS Z-0208 / ASTM E-96

PP as OUTER LAYER (after retort)
- PP / Tie / PA / Tie / PP
- PP / Tie / PA / Tie / PP

PA as OUTER LAYER (after retort)
- PA / EVOH / PA / Tie / PP
- PP / Tie / PA / EVOH / PA
Moisture dependence of NYLON

UBE NYLON COPOLYMER
@ 23 °C, RH 50%

“Wet”: HIGHER WVTR, QUICK DRY

“Dry”: LOWER WVTR, PROTECTION

“WET” conditions: ASTM E-96 Water method
“DRY” conditions: ASTM E-96 Desiccant method

Retort packaging
### Film structures

<table>
<thead>
<tr>
<th>Symmetrical structures</th>
<th>Asymmetrical structures</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Conventional</strong></td>
<td></td>
</tr>
<tr>
<td>Medium barrier</td>
<td></td>
</tr>
<tr>
<td>PP / Tie / PA / Tie / PP</td>
<td>PA / Tie / PP / PP / PP</td>
</tr>
<tr>
<td>High barrier</td>
<td></td>
</tr>
<tr>
<td>PP / Tie / EVOH / Tie / PP</td>
<td>PA / EVOH / PA / Tie / PP</td>
</tr>
<tr>
<td>PP / Tie / PA / EVOH / PA / Tie / PP</td>
<td></td>
</tr>
</tbody>
</table>

For the study:
- Total film thickness: 100μm
- PA layer thickness: 30%
- EVOH layer thickness: 10%
### Oxygen Transmission Rate (by OTR)

<table>
<thead>
<tr>
<th>Symmetrical structures</th>
<th>Asymmetrical structures</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Conventional</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Medium barrier</strong></td>
<td><strong>High barrier</strong></td>
</tr>
<tr>
<td>PP / Tie / PA / Tie / PP</td>
<td>PA / Tie / PP / PP / PP</td>
</tr>
<tr>
<td>Analysis time</td>
<td>Analysis time</td>
</tr>
<tr>
<td>(OTR stabilization)</td>
<td>(OTR stabilization)</td>
</tr>
<tr>
<td>Before retort</td>
<td>Before retort</td>
</tr>
<tr>
<td>After retort</td>
<td>After retort</td>
</tr>
<tr>
<td>14 days</td>
<td>4 days</td>
</tr>
</tbody>
</table>

**Autoclave conditions**: $T=125^\circ C$, $t=30\text{min}$  
**Room conditions**: $T=23^\circ C$, RH $=50\%$ $t=24\text{h}$  
**Testing conditions**: $T=23^\circ C$, RH $\left(O_2\right) =50\%$ - RH $\left(N_2\right) = 90\%$

- Films don’t recover 100% initial $O_2$ barrier after retort treatment
- Faster drying = Faster barrier recovery in asymmetrical structures
Agenda

- Retort – Process & Food
- O₂ Barrier vs moisture
- New retort packaging concept
- Evaluation methodology & Data
- Which UBE NYLON for retort?
- Case study
- Conclusions
1- Pouches preparation:
   - Tubular film (only 2 sealing of the pouch)
   - Always same dimensions

2- Sensors fixed in the packages:
   - For the non-invasive optical measurement
Evaluation methodology

3- Tomato sauce filling:
   - Always same sauce and same amount

4- Introduction of temperature and pressure recorder:
Evaluation methodology

5- Pouches closing:
- Vacuum
- Introduction of $N_2$ in the head space
- Sealing

6- Autoclave treatment:
Evaluation methodology

Autoclave conditions:

\[ T = 121\, ^\circ C \]
\[ t = 30 \, \text{min} \]
Evaluation methodology

7- Storage conditions:
   - Temperature: \( T = 23^\circ C \),
   - Relative humidity: \( RH = 50\% \),
   - Time: \( t = \) up to 6 months
Oxygen Permeation (optical measurement)
Oxygen Permeation (optical measurement)

Oxygen permeation after retort treatment

- **Sym. Med. Barrier**: PP / Tie / PA / Tie / PP
- **Sym. High. Barrier**: PP / Tie / EVOH / Tie / PP
Oxygen Permeation (optical measurement)

Oxygen permeation after retort treatment

- **Sym. Med. Barrier**
  - PP / Tie / PA / Tie / PP

- **Asym. Med. Barrier**
  - PA / Tie / PP / PP / PP

- **Sym. High. Barrier**
  - PP / Tie / EVOH / Tie / PP

Time after retort treatment (h)
Oxygen Permeation (optical measurement)

Oxygen permeation after retort treatment

- Sym. Med. Barrier: PP / Tie / PA / Tie / PP
- Sym. High. Barrier: PP / Tie / EVOH / Tie / PP
- Asym. High barrier: PA / EVOH / PA / Tie / PP
Agenda

- Retort – Process & Food
- O₂ Barrier vs moisture
- New retort packaging concept
- Evaluation methodology & Data
- Which UBE NYLON for retort?
- Case study
- Conclusions
Which UBE NYLON?

Structure:

PA/Tie/PA/Tie/PP or PA/EVOH/PA/Tie/PP

Outer layer:

HYDROLISIS Resistant - UBE NYLON 5033FD8

Tested up to T=125°C, t=3h

PA hydrolysis reaction

50 µm monolayer airblown film

UBE NYLON 5033B

UBE NYLON 5033FD8

Retort packaging
Which UBE NYLON?

Structure:

- PA/Tie/PA/Tie/PP
- PA/EVOH/PA/Tie/PP
- PP/Tie/PA/Tie/PP

Middle layer:

**Autoclave treatment at T=125ºC**

- **Moderate conditions (up to 2h):**
  - UBE NYLON 1030B
  - UBE NYLON 5033B
  - UBE NYLON 5033FD8

- **Severe conditions (more than 2h):**
  - UBE NYLON 5033FD8
Agenda

- Retort – Process & Food
- O₂ Barrier vs moisture
- New retort packaging concept
- Evaluation methodology & Data
- Which UBE NYLON for retort?
- Case study
- Conclusions
Application in carrot puree - Methodology

Carrots peeling

Carrots cutting

Carrots cooking: $T = 100^\circ C$
$t = 45$ min

Carrots mashing

Recipe preparation. Addition:
- 10% water
- Citric acid until pH = 5.2

Recipe preparation

Packing and sterilization:
- $T = 121^\circ C$
- $t = 15$ min
- $P = 2.2$ bar

Retort packaging
Application in carrot puree - Evaluation

Conditioning (storage):
- Standard: $T = 20^\circ C$
- Accelerated: $T = 40^\circ C$

Parameters:
- Color change $\Delta E$

<table>
<thead>
<tr>
<th>$\Delta E$</th>
<th>Perception of change</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 0.2</td>
<td>Not perceptible</td>
</tr>
<tr>
<td>0.2 – 0.5</td>
<td>Perceptible through close observation</td>
</tr>
<tr>
<td>0.5 – 1.5</td>
<td>Perceptible</td>
</tr>
<tr>
<td>1.5 – 3.0</td>
<td>Perceptible at a glance</td>
</tr>
<tr>
<td>3.0 – 6.0</td>
<td>Obvious</td>
</tr>
<tr>
<td>6.0 – 12.0</td>
<td>Significant</td>
</tr>
<tr>
<td>&gt; 12.0</td>
<td>Very significant</td>
</tr>
</tbody>
</table>

CIE Lab
Application in carrot puree - Color

Standard Temperature (T=20ºC)

@ Standard Temperature (T=20ºC)

- Sym. High barrier: PP / Tie / EVOH / Tie / PP
  - $y = 0.2252x$
- Asym. Medium barrier: PA / Tie / PP / PP / PP
  - $y = 0.2242x$
- Asym. High barrier: PA / EVOH / PA / Tie / PP
  - $y = 0.0483x$

Storage/Conditioning time (days) vs. $\Delta E$
Application in carrot puree - Color

Standard Temperature (T=20ºC)

Sym. High barrier: PP / Tie / EVOH / Tie / PP
Asym. Medium barrier: PA / Tie / PP / PP / PP
Asym. High barrier: PA / EVOH / PA / Tie / PP

Retort packaging
Application in carrot puree - Color

Accelerated Temperature (T=40ºC)

\[ y = 0.1263x \]

Storage/Conditioning time (days)

@ Standard Temperature (T=40ºC)

Sym. High barrier
PP / Tie / EVOH / Tie / PP

Asym. Medium barrier
PA / Tie / PP / PP / PP

Asym. High barrier
PA / EVOH / PA / Tie / PP
Application in carrot puree - Color

Accelerated Temperature (T=40ºC)

Sym. High barrier
PP / Tie / EVOH / Tie / PP

Asym. Medium barrier
PA / Tie / PP / PP / PP

Asym. High barrier
PA / EVOH / PA / Tie / PP

Retort packaging
Application in carrot puree - Shelf life

Criteria: Color change not accepted by consumer (ΔE = 29)

Calculated Shelf life:

- **Sym. High barrier**
  - PP / Tie / EVOH / Tie / PP
  - 127 - 134 days
  - 4 to 4.5 months

- **Asym. Medium barrier**
  - PA / Tie / PP / PP / PP
  - 128 - 136 days
  - 4 to 4.5 months

- **Asym. High barrier**
  - PA / EVOH / PA / Tie / PP
  - 308 - 340 days
  - 10 to 11 months

- Oxygen permeation results could be extrapolated to shelf life
- New concept increases more than two times shelf life of the product
Agenda

Retort – Process & Food

O₂ Barrier vs moisture

New retort packaging concept

Evaluation methodology & Data

Which UBE NYLON for retort?

Case study

Conclusions
Conclusions

1) **Medium barrier asymmetrical films (PA/Tie/PP/PP/PP)**: Good alternative to current high barrier symmetrical films (PP/Tie/EVOH/Tie/PP). Besides they offer superior mechanical and optical properties.

2) **High barrier asymmetrical films (PA/EVOH/PA/Tie/PP)**: Solution to extend significantly the shelf life of the foodstuffs. Besides they offer higher mechanical and optical properties.

3) **UBE NYLON 5033FD8** (unique material in the market) offers the possibility to place PA in outer layer of retort packaging.
Thank You

very much for your attention

Sergi Salvà Sàez
Technical Development Manager
UBE America Inc.
E-mail: s.salva@ube.com