Shelf-life study of freeze dried ‘sambal tumis bilis’
(Kajian tentang jangkamasa simpan sambal tumis bilis kering beku)

H. A. Hasimah*, M. K. Noraini* and M. Y. Rafiah Hasanah*

Key words: freeze dried ‘sambal tumis bilis’, packaging, sensory, shelf-life

Abstract
’Sambal tumis bilis’, a popular Malaysian dish of fried anchovies in chilli gravy, was freeze dried to 0.73% moisture. Freeze dried sambal tumis bilis samples packed in polyester/aluminium/polyethylene (PET/Al/PE) and polyester/polyethylene/aluminium/polyethylene/special film (PET/PE/Al/PE/SF) with and without nitrogen flushing, were stored for 8 months at 25 °C. Free fatty acid determination, sensory and microbiological evaluations were conducted to determine the quality of the products. Without nitrogen flushing, freeze dried sambal tumis bilis packed in both packaging materials had a shelf-life of 3 months while those packed with nitrogen had a shelf-life of at least 8 months. Nitrogen flushing was thus able to extend the shelf-life of the product for another 5 months.
Shelf-life of ‘sambal tumis bilis’

The packaging material should have good barrier properties to the factors mentioned. Both vacuum and gas packaging can be used to eliminate oxygen from the package to reduce the occurrence of oxidative rancidity since oxygen levels fall to 1–2% (Sacharow and Griffin 1980). The effectiveness of nitrogen flushing in extending shelf-life of various products packed in various types of packaging has been reported (Anon. 1985; Rice 1986).

This study was undertaken to determine the suitability of two types of packaging material for packing and storing freeze dried sambal tumis bilis, and also to determine the effect of nitrogen flushing on product shelf-life.

Materials and methods

Preparation of freeze dried sambal tumis bilis
Dried chillies, shallots, garlic and prawn paste were blended and fried in vegetable oil. Sugar, salt, tamarind juice and water were then added. Cooking was continued until the gravy was thick into which fried dried anchovies were added. The cooked product was put into stainless steel trays and frozen at –25°C for 18 h before drying at 65.5°C for 7 h at a pressure of 1 torr using a freeze dryer (Vacudyne Model VPFD-A, USA).

For this study, a pool of freeze dried samples were prepared, packed and stored. Samples were taken from this pool for analysis at regular intervals.

Packaging
Freeze dried sambal tumis bilis (25 g) was packed in 8 cm x 13 cm laminated pouches of polyester/aluminium foil/polystyrene (PET/Al/PE) of 12 gsm : 9 µ : 75 gsm combination and polyester/polystyrene/aluminium foil/polystyrene/special film (PET/PE/Al/PE/SF) of 12 µ : 20 µ : 7 µ : 20 µ : 60 µ thickness (Alcon Packaging). Half of the samples of each type of packaging material were flushed with 40% nitrogen while the remainder were not. This was immediately followed by heat sealing. Hence, the four treatments evaluated were:
- PET/Al/PE without nitrogen,
- PET/PE/Al/PE/SF without nitrogen,
- PET/Al/PE with nitrogen, and
- PET/PE/Al/PE/SF with nitrogen,

The samples were then stored at 25 °C, 65% r.h. for 8 months.

Analysis and evaluation
Chemical analysis of free fatty acid (FFA) and sensory evaluation were conducted monthly during the 8 months of storage. Microbiological evaluation was conducted at 0, 4 and 8 months of storage. Proximate analysis was conducted on the fresh sambal tumis bilis and the freeze dried product before packing.

Chemical analysis  For each treatment, three pouches of product were taken from the sample pool, mixed and analysed. Proximate analysis (in duplicate) was determined according to Pearson (1976). FFA determination (in duplicate) was done according to the method of AOAC (1984) and results were expressed as oleic acid.

Microbiological analysis  A 10 g sample was homogenised with 90 mL of one-quarter strength Ringers solution for 2 min using a Seward Stomacher. Serial dilutions were made using 9 mL of liquid from the homogenate. The appropriate serial dilutions were used for the determinations of total viable count, yeasts and moulds count, coliform count, Staphylococcus aureus count and Escherichia coli count using methods recommended by ICMSF (1978). Water activity (a_w) was determined at 23–25 °C with a Rotronic Hygroskop DT model meter model CH8040, Zurich.

Sensory evaluation  The packed products and a control of freshly prepared freeze dried sample were evaluated organoleptically at monthly intervals. For each treatment, five pouches of product
were taken from the sample pool, mixed and rehydrated. Each pouch of freeze dried sample (25 g) required 25 mL of boiling water for rehydration. Twenty taste panelists assessed the samples for overall acceptability which covered factors such as taste, texture, aroma and colour. Sensory evaluation scores were described by using a 9-point hedonic rating scale ranging from 1 (dislike extremely) to 9 (like extremely) (Larmond 1977). Panelists were also asked if rancidity was detectable. Samples flushed with nitrogen were assessed separately from those packed without nitrogen flushing.

**Statistical analysis**  The average score of overall acceptability was used as a measure of product acceptance. A two-way analysis of variance (ANOVA) was conducted on the samples packed without nitrogen throughout the 8-month storage period to determine if the difference between the treatments was significant. This was repeated for samples flushed with nitrogen. Those shown to be significant by ANOVA were further tested using Least Significant Difference (LSD) test to estimate which average scores were statistically different (Larmond 1977).

### Table 1. Proximate composition of sambal tumis bilis

<table>
<thead>
<tr>
<th>Composition</th>
<th>Freshly cooked</th>
<th>Freeze dried</th>
</tr>
</thead>
<tbody>
<tr>
<td>Protein (%) (N x 6.25)</td>
<td>19.94</td>
<td>38.60</td>
</tr>
<tr>
<td>Fat (%)</td>
<td>22.24</td>
<td>37.80</td>
</tr>
<tr>
<td>Ash (%)</td>
<td>3.96</td>
<td>7.35</td>
</tr>
<tr>
<td>Moisture (%)</td>
<td>45.80</td>
<td>0.73</td>
</tr>
</tbody>
</table>

**Results and discussion**

**Proximate analysis**

The main ingredient of fried anchovies accounted for the fairly high protein and fat content (*Table 1*). The moisture content of freeze dried sambal tumis bilis was very low (0.73%) compared with the freshly cooked product (45.80%). The high loss in moisture due to the freeze drying process resulted in the concentration of the other constituents. The high fat and low moisture content of the freeze dried product indicated the possibility of the product becoming rancid and absorbing moisture if the packaging materials chosen were not suitable.

**Free fatty acid**

The FFA values for all treatments during the 8-month storage were fairly stable in the range of 0.35–0.69% oleic acid despite the higher values initially (*Figure 1*). Rancidity
Shelf-life of ‘sambal tumis bilis’ is usually accompanied by FFA formation and its determination can be used to indicate the condition and edibility of oils. For most oils, the threshold for acidity to be detected by the palate is when FFA is about 0.5–1.5% oleic acid, whereas for beef with 10.6–25.1% fat, FFA should not exceed 1.2% oleic acid (Egan et al. 1981). The freeze dried sambal tumis bilis had a fat content of 37.80% and a maximum FFA value of 0.69% oleic acid. At these levels, the taste panelists did not detect rancidity in the samples when questioned in the sensory evaluation forms.

The packaging treatments used were effective in preventing oxidation of the products up to 8 months. The combination of aluminium, polyester and polyethylene used in the packaging material laminates gave good barrier to oxygen (Cox 1968).

**Microbiological evaluation**

All the treatments were microbiologically acceptable as counts were < 3.0 x 10^2 colony forming units per gram (cfu/g) sample at 0, 4 and 8 months of storage (Table 2). Coliforms, *Staphylococcus aureus* and *Escherichia coli* were not detected in all samples. Both packaging materials, PET/Al/PE and PET/PE/Al/PE/SF, were successful in preventing moisture uptake and caking of the product since a_w of the product in the four treatments were fairly constant. This was consistent with the findings of Cox (1968), and Sacharow and Griffin (1980) who reported that polyethylene had good barrier properties to moisture vapour transmission while aluminium was impermeable to moisture vapour.

Flushing the packed products with nitrogen did not cause any increase in the microbiological counts during the 8-month storage period. Similar observation was reported by Pinto (1979) who noted that nitrogen inhibited the growth of aerobic bacteria.

**Table 2. Microbiological counts of freeze dried sambal tumis bilis during storage**

<table>
<thead>
<tr>
<th>Storage time (months)</th>
<th>Treatment</th>
<th>Total viable count (cfu/g)</th>
<th>Yeasts and moulds count (cfu/g)</th>
<th>a_w at 23–25 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>PET/Al/PE</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Without nitrogen</td>
<td>&lt; 3.0 x 10^2 (2.0 x 10)</td>
<td>&lt; 1.0 x 10</td>
<td>0.13</td>
</tr>
<tr>
<td></td>
<td>With nitrogen</td>
<td>&lt; 1.0 x 10</td>
<td>&lt; 1.0 x 10</td>
<td>0.12</td>
</tr>
<tr>
<td></td>
<td>PET/PE/Al/PE/SF</td>
<td>&lt; 3.0 x 10^2 (6.0 x 10)</td>
<td>&lt; 1.0 x 10</td>
<td>0.13</td>
</tr>
<tr>
<td></td>
<td>Without nitrogen</td>
<td>&lt; 1.0 x 10</td>
<td>&lt; 1.0 x 10</td>
<td>0.13</td>
</tr>
<tr>
<td></td>
<td>With nitrogen</td>
<td>&lt; 1.0 x 10</td>
<td>&lt; 1.0 x 10</td>
<td>0.13</td>
</tr>
<tr>
<td>4</td>
<td>PET/Al/PE</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Without nitrogen</td>
<td>&lt; 1.0 x 10</td>
<td>&lt; 1.0 x 10</td>
<td>0.14</td>
</tr>
<tr>
<td></td>
<td>With nitrogen</td>
<td>&lt; 1.0 x 10</td>
<td>&lt; 1.0 x 10</td>
<td>0.12</td>
</tr>
<tr>
<td></td>
<td>PET/PE/Al/PE/SF</td>
<td>&lt; 1.0 x 10</td>
<td>&lt; 1.0 x 10</td>
<td>0.14</td>
</tr>
<tr>
<td></td>
<td>Without nitrogen</td>
<td>&lt; 1.0 x 10</td>
<td>&lt; 1.0 x 10</td>
<td>0.13</td>
</tr>
<tr>
<td></td>
<td>With nitrogen</td>
<td>&lt; 1.0 x 10</td>
<td>&lt; 1.0 x 10</td>
<td>0.13</td>
</tr>
<tr>
<td>8</td>
<td>PET/Al/PE</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Without nitrogen</td>
<td>&lt; 3.0 x 10^2 (4.0 x 10)</td>
<td>&lt; 1.0 x 10</td>
<td>0.14</td>
</tr>
<tr>
<td></td>
<td>With nitrogen</td>
<td>&lt; 1.0 x 10</td>
<td>&lt; 1.0 x 10</td>
<td>0.14</td>
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<td>&lt; 1.0 x 10</td>
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<tr>
<td></td>
<td>With nitrogen</td>
<td>&lt; 1.0 x 10</td>
<td>&lt; 1.0 x 10</td>
<td>0.13</td>
</tr>
</tbody>
</table>
Sensory evaluation

The average scores for overall acceptability of the freshly freeze dried and stored samples packed in PET/Al/PE and PET/PE/Al/PE/SF without and with nitrogen flushing are shown in Figure 2. Scores >5.0 are taken as being acceptable to the panelists. Shelf-life, as used in this context, referred to the period during which the quality of the test product remained the same as the fresh product, whereas an acceptable product meant that the quality may have deteriorated but was still tolerable unless proven that the product was no longer wholesome.

Samples packed without nitrogen flushing in both types of packaging material had the same quality as the freshly prepared sample up to 3 months of storage. Differences in quality were observed by the fourth month of storage. However, the products were still acceptable to the panelists even up to 7 months of storage. This was reflected by the average overall acceptability scores which were greater than 5.0 and rancidity which was not detected by the panelists (Table 3). By the eighth month, both products were unacceptable to the panelists. Thus the shelf-life of products packed in both packaging materials without nitrogen flushing was 3 months and no significant difference was observed between the two types of packaging material.

Products packed in PET/Al/PE and PET/PE/Al/PE/SF with nitrogen flushing were still acceptable after 8-month storage. The product stored in PET/Al/PE for 8 months, however, was significantly different from PET/PE/Al/PE/SF-stored product which obtained higher scores (Table 3). This indicated that PET/PE/Al/PE/SF was more suitable for long-term storage of freeze dried sambal tumis bilis with nitrogen flushing. A comparison of freshly prepared freeze dried sample with both nitrogen flushed freeze dried sample in the two packaging materials showed that there were no significant differences among the three samples after 8 months.

The effect of nitrogen flushing can be observed by the extension of shelf-life of freeze dried product packed in PET/Al/PE and PET/PE/Al/PE/SF by 5 months.

Figure 2. Average score for overall acceptability of freeze dried sambal tumis bilis packed without and with nitrogen flushing
Table 3. Least significant difference test on freeze dried sambal tumis bilis packed without and with nitrogen flushing

<table>
<thead>
<tr>
<th>Storage time (months)</th>
<th>Av. score for product overall acceptability, without nitrogen</th>
<th>Av. score for product overall acceptability, with nitrogen</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>A</td>
<td>B</td>
</tr>
<tr>
<td>0</td>
<td>7.10</td>
<td>–</td>
</tr>
<tr>
<td>1</td>
<td>6.75a</td>
<td>5.90a</td>
</tr>
<tr>
<td>2</td>
<td>6.25a</td>
<td>6.05a</td>
</tr>
<tr>
<td>3</td>
<td>6.15a</td>
<td>6.10a</td>
</tr>
<tr>
<td>4</td>
<td>6.80a</td>
<td>5.25b</td>
</tr>
<tr>
<td>5</td>
<td>7.15a</td>
<td>5.60b</td>
</tr>
<tr>
<td>6</td>
<td>7.05a</td>
<td>5.85b</td>
</tr>
<tr>
<td>7</td>
<td>7.00a</td>
<td>5.10b</td>
</tr>
<tr>
<td>8</td>
<td>6.35a</td>
<td>4.40b</td>
</tr>
</tbody>
</table>

Average scores within the same row with the same letter for the same treatment are not significantly different (p < 0.05)

A = freshly freeze dried sample
B = stored sample in polyester/aluminium/polyethylene
C = stored sample in polyester/polyethylene/aluminium/polyethylene/special film

**Conclusion**

Without nitrogen flushing, the shelf-life of freeze dried sambal tumis bilis packed in PET/Al/PE and PET/PE/Al/PE/SF was 3 months while those packed with nitrogen had a shelf-life of at least 8 months at 25 °C. Nitrogen flushing extended the shelf-life of freeze dried sambal tumis bilis by 5 months for both PET/Al/PE and PET/PE/Al/PE/SF while retaining the sensory qualities of the fresh product.

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**References**


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