

Shelf-life model: Useful tool to predict sensory and nutritional quality of infant formulas

**CHRISTINE KERSCH-COUNET¹, Petra Vossenber¹, Rudy de Wit¹,
Wim Timmermans¹, Hans Cruijse¹, Jaganmohan Rallapalli² and Paul Deckers²**

¹ *FrieslandCampina Nederland B.V., Stationsplein 4, 3818 LE Amersfoort, The Netherlands*

² *Bodec Process Technology B.V., Scheepsboulevard 3, 5705 KZ Helmond, The Netherlands*

Abstract

Shelf life prediction gains an increasing interest in food industries. It is especially relevant for long shelf-life products where degradations have more time to occur. In this study, we show that it is possible to build a reliable shelf-life kinetics model for infant formula (powder) packed in metal cans. The model varies the following parameters: storage temperature (5-40°C), storage time (0-2 years), and oxygen level in the pack (protected or unprotected atmosphere). The effects of light and moisture were discarded as they cannot penetrate through the metal can. A model was build based on chemical kinetics. The model is able to predict the taste, the level of vitamin C, and the aromas concentrations based on the chemical reactions occurring in the infant formula. The kinetic reactions were fitted based on data of aroma concentrations and oxygen level in the package. Several examples of accelerated shelf life tests simulating a normal shelf life at 2 years are illustrated. The results are compared to the most common practice in shelf-life: using a fixed Q10 temperature coefficient. It is advised to use multiple accelerated shelf life tests to mimic the normal shelf life of the relevant sensory or nutritional aspects of the product.

Introduction

In this study, we show that it is possible to build a reliable shelf-life kinetics model for infant formula (powder) packed in metal cans.

Experimental

Shelf-life conditions

In order to build the model several conditions were varied in the infant formula:

- Storage temperature: 5°C, 20°C, 30°C, and 40°C,
- Storage time: 0 to 2 years,
- Oxygen level (O₂) in the pack: protected (N₂ flushed) or unprotected atmosphere (21 % O₂).

The effects of light and moisture were discarded in this study as they cannot penetrate through the metal can [1, 2, 3].

Analysis

Several selected parameters have been measured in the infant formula (powders):

- Sensory attributes (Quantitative Descriptive Analysis - QDA, scale 0-100) were evaluated by a trained panel of 16 persons,
- Aromas concentration was determined by GC-MS (most relevant aromas selected based on literature [4] and internal check; method adapted from [5]),
- Oxygen content in the package (metal can) and vitamin C content.

Model building

The relevant correlations between the parameters mentioned above were identified using multivariate analysis methods (Unscrambler). The gPROMS model builder was used to calculate the kinetics of the chemical reactions (aromas, vitamin; [6]). A user-friendly shelf-life model was finally created in Excel and linked to the gPROMS model builder interface. The predictive power of the model was validated with real data. Utilizing this model, sensory attributes scores or vitamin C level can be predicted based on aroma compounds or/and oxygen evolution in the package during storage.

Results and discussion

General trends

During shelf-life, infant formula powders were very sensitive to oxygen exposure. This effect is even more prevalent if the temperature increased during storage. As an example (Figure 1a), an infant formula packed in a metal can without protected atmosphere (high level of oxygen) developed higher oxidation flavour and showed high losses of vitamin C during storage. These results were expected as vitamin C is known to be one of the most unstable vitamins to oxygen and heat [7]. Similar results were obtained for liquid dairy products where other vitamins (B1, B2, D or A) always showed less degradation than vitamin C during storage with oxygen (data not shown).

In contrast to unprotected atmosphere, infant formula powders packed in the metal can with protected atmosphere (low oxygen level) were extremely well protected. Vitamin C was stable at any temperature tested (30-60°C) and only a slight increase (not significant) of oxidation flavour occurred after 2 years storage for the common temperatures of 30-40°C (end of shelf-life; Figure 1b). At 60°C, the oxidation reactions with the residual O₂ content were increased.

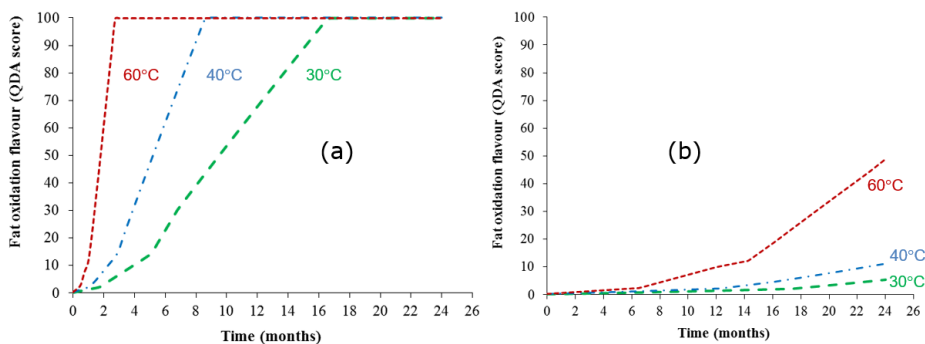


Figure 1: Increase of fat oxidation flavour with storage time (months) for (a) an infant formula in a metal can containing 21% O₂; (b) an infant formula in a metal can flushed with inert gasses (protected atmosphere; 1.5% O₂ residual).

Fitting with aroma compounds

A very good fitting was identified between the fat oxidation flavour and several aromas for the infant formula (correlation >0.6, good fitting in the model): hexanal (impact ~60%), pentanal (impact ~ 25%), 2,4-tr, tr-decadienal (impact <5%), 4-cis-heptenal (impact < 5%), penten-3-one (impact < 5%). Furthermore, furfural fitted with the burned odour observed during storage in the dried infant formula powder. Such a result is logical as furfural is produced through Maillard reactions [8]. Those reactions

trend to happen during storage at high temperature and can increase the caramelized, sweet or burned notes of the product. During storage, production of furfural in infant formula was only linked to heat state and not to the oxygen level. Though it is mainly recognized that Maillard reactions increase in the presence of oxygen [8] some authors however underlined that those reactions can also occur in anaerobic conditions [9]. The latter statement is supported by the finding that browning reactions were also observed during storage in dairy drinks in anaerobic conditions (data not shown).

Q10 method

A tool commonly used in accelerated shelf-life studies is the Q10 method. Q10 is the factor that indicates the increase in the rate of the reactions when the temperature is increased by 10°C. It is unit less and can be calculated with the following equation for 2 reactions 10°C apart: $Q10 = k(T+10^\circ\text{C}) / k(T^\circ\text{C})$, where k = reaction rate constant. For most products, the Q10 value is 2.0, which means for every increase of 10°C, the rate of a chemical reaction will double. As an example, if a food has a stability of 20 weeks at 20°C and 10 weeks at 30°C, then the Q10 will be 20/10 or 2.

Accelerated shelf-life tests simulating a normal shelf-life at 2 years

As it can be seen from Tables 1-2, the model was used to predict the accelerated shelf life test to mimic a normal storage of 2 years at 30°C of infant formula packed in a metal can. The results were compared with the common approach, i.e. the Q10 method (see description above).

Table 1: Accelerated shelf life of infant formula mimicking the values obtained after 2 years at 30°C in a metal can flushed with inert gasses (1.5% O₂ residual in the headspace).

	Value after 2 years at 30 °C*	Corresponding months at 40 °C	Corresponding months at 60 °C [#]	Equivalent Q10 30 °C vs 40 °C
Vitamin C (mg/kg)	873	12.3	3.3	2.0
Fat oxidation flavour	5	16.5	8.3	1.5
Hexanal (ppb) - A	254	15.8	7.3	1.5
Furfural (ppb) - B	60	5.0	0.3	4.8

* Initial value before storage: vitamin C: 890 mg/kg; fat oxidation flavour:1; hexanal: 10 ppb; furfural: 20 ppb. Indicator of oxidation reactions (A) or of Maillard reactions (B). [#] Values at 60°C were generated by the shelf-life prediction model.

Table 2: Accelerated shelf life of infant formula mimicking the values obtained after 2 years at 30°C for a metal can in unprotected atmosphere conditions (21% O₂ in the headspace).

	Value after 2 years at 30 °C*	Corresponding months at 40 °C	Corresponding months at 60 °C [#]	Equivalent Q10 30 °C vs 40 °C
Vitamin C (mg/kg)	738	10.3	2.2	2.3
Fat oxidation flavour	100	8.8	3.0	2.7
Hexanal (ppb) - A	14992	12.5	4.0	1.9
Furfural (ppb) - B	60	5.0	0.3	4.8

* Initial value before storage: vitamin C: 890 mg/kg; fat oxidation flavour:1; hexanal: 10 ppb; furfural: 20 ppb. Indicator of oxidation reactions (A) or of Maillard reactions (B). [#] Values at 60°C were generated by the shelf-life prediction model.

The shelf-life parameters of the infant formula (vitamin C, fat oxidation, hexanal, furfural) showed different kinetics and therefore, they should be tested using different accelerated shelf-life (Table 1). For example, the furfural (indicator of Maillard reactions) needed an accelerated shelf-life at 40°C of 5 months to mimic the normal shelf life of 2

years at 30°C while the fat oxidation needs 16.5 months. This also means that the Q10 coefficient of the different parameters varied from 1.5 to 4.8. Only one factor has a Q10 coefficient of 2 in a metal can with protected atmosphere: the vitamin C. The Q10 factor can vary in function of the conditions, for example a Q10 of 1.5 was observed for fat oxidation in protected atmosphere (Table 1) while it was closer to 3 in unprotected atmosphere at 40°C (Table 2). The accelerated shelf-life of the infant formula at 60°C showed that all reactions can be accelerated but that for several parameters still some months were required to reach the same value found in normal shelf-life (2 years at 30°C). As an example, an accelerated shelf-life of 7.3 months and 2.8 months, both at 60°C were needed for fat oxidation and vitamin C, respectively (Table 1). This is logical since metal cans with protected atmosphere are extremely good protective packaging [2, 3].

The same observations were seen in unprotected atmosphere conditions: the accelerated shelf-life conditions (Table 2) as well as the Q10 coefficient depended on the parameter types (vitamin C, fat oxidation). The fat oxidation flavour as well as oxidation reactions indicator, hexanal increased sharply in unprotected conditions (see values of normal shelf life at 30°C for 2 years Table 1 vs Table 2). The Maillard reactions indicator (furfural) was similar at 30°C for protected and unprotected atmosphere. This is because those reactions were dependent on the applied temperature and not on the oxygen level present in the headspace of the packaging.

The results indicate that using only one accelerated shelf-life test (one time/temperature) to mimic the normal shelf-life is not optimal. The best approach would be to use one accelerated shelf-life test for each parameter of interest. In other words, a multiple shelf-life approaches should be used, respecting the reaction kinetic of each parameter. In the near future, shelf-life model will help to better predict the behaviour of the key parameters of infant formula powders and to correlate the results to normal shelf life. With this model, the duration of accelerated shelf life study is expected to reduce while still guarantying a good prediction of the normal shelf-life.

References

1. Robertson, G. L. (2009). In *Food packaging and shelf-life: a practical guide* (Robertson, G. L., ed). CRC Press, USA. pp 1-16.
2. Gargouri, B., Zribi, A., Bouaziz, M. (2015). *J Food Sci Technol*, 52: 1948–1959.
3. Properties of packaging material: <http://www.fao.org/Wairdocs/X5434E/x5434e0g.htm>.
4. Venkateshwarlu, G., Let, M. B., Meyer, A. S., Jacobsen, C. (2004). *J. Agric. Food Chem*, 52: 311-317.
5. Jeleň, H.H., Dabrowska, A., Klensporf, D., Nawrocki, J., Wasowicz, E. (2004). *Chem. Anal*, 49: 869.
6. Van Boekel, T. (2010). In *Analysis in Controlling Maillard Pathways to Generate Flavors* (Mottram and Taylor, eds), ACS Symposium: Washington, DC: pp. 1-11.
7. Paulus, K. (1989). *Bibliotheca Nutritio et Dieta*, 43 : 173–187.
8. Mottram, D. S. (2007). In *Flavours and Fragrances* (Berger, R.G., ed.), Springer Verlag Berlin Heidelberg, pp. 269-283.
9. Hayase, F., Shibuya, T., Sato, J., Yamamoto, M. (1996). *Biosci Biotechnol Biochem*. 60: 1820-5.