Evaluation of shelf-life of fruit baby food

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Abstract. Fruit baby food is an important food source in infant nutrition. This ambient stable product is processed using heat treatment and can be stored for one or more years at ambient temperature. An accelerated shelf-life storage test of fruit baby food was carried out. Sets of samples were stored at various storage temperatures (40, 55, 70 and 90 °C) for 3 weeks. Selected markers were followed and correlated with sensory evaluation during the storage. The markers were: DPPH, total phenols, ascorbic acid, 5-HMF, furfural and colour (expressed as L, a*, b* and ΔE). Kinetic data (reaction rate constants, activation energies, Q_10, z values) were calculated. The aim of the paper was to evaluate shelf-lives of fruit baby food. The colour parameters, especially ΔE, seem to be a robust criterion which could be used to predict shelf-lives of fruit baby food.

Key words: kinetics, modelling, storage experiment, colour.

INTRODUCTION

Determination of storage periods is one of the most important steps in food production. A shelf-life is defined as a period during which food products meet specific criteria. Food safety is the key criterion that must be fulfilled. Approach to determination of storage periods depends on a type of food product (Fu & Labuza, 1993). Microbial growth is an important criterion in case of perishable foods (Dalgaard, 1995; Corbo et al., 2006). In case of imperishable foods, selected parameters are associated with nutritionally important compounds, such as vitamins, antioxidants etc., or with changes of sensory properties of food (especially colour and texture). Conventional storage tests can be used to estimate storage periods; during such tests, food is stored for a long time under conditions which are expected during normal storage of the particular commodity. Another option for estimating storage periods is an accelerated storage test. Appropriate markers are then chosen on the basis of a literature review.

An accelerated shelf-life test (ASLT) is carried out for several selected markers; during the test, a particular foodstuff is stored for a specific period of time at higher temperatures or exposed to higher concentrations of oxygen etc. (Ragnarsson & Labuza, 1997; Hough et al., 2006; García-Garcia et al., 2008). Changes of the markers are evaluated by ordinary kinetic procedures. The choice of a reaction order of the chosen markers may be based on literature research. If relevant data has not been published, it is necessary to determine a reaction order experimentally. Determination of a reaction order is governed by different types of algorithms and testing by kinetic modelling software (Sande & Karlse, 1993; Larsson & Pardue, 1990). The key point of shelf-life
determination is to estimate limits of the selected markers. Sensory markers are the most important parameters for shelf-life prediction. The Weibull model and some others (e.g. Markov model) are the most widely used tools for determination of sensorial limits (Freitas & Costa, 2006; Ledauphin et al., 2006; Palazón et al., 2009).

Commercial fruit baby food is a preserved fruit product usually made of fruit purees, sugar, water and different additives (thickening agents, antioxidants, etc.), which is aseptically bottled and pasteurized and then subjected to another heat treatment and pasteurization; finally the final product is put into long-time storage. As a foodstuff intended for specific nutritional uses, baby foods for infants and young children conform to a set of strict guidelines, e.g. maximum levels for pesticide residues, microbiological contamination, addition of additives, labelling, etc. (Čižková et al., 2009).

Evaluation of changes of chemical markers is important for prediction of shelf-lives of fruit-based products. Sucrose isomerisation (Opatová et al., 1992), formation of 2-furaldehyde and 5-hydroxymethyl furfural (Rada-Mendoza et al., 2002; Burdurlu & Karadeniz, 2003; Gentry & Roberts, 2004), furosine (Rada-Mendosa et al., 2004), levulenic acid (Opatová et al., 1992) and degradation of ascorbic acid (Palazón et al., 2009) etc. are the principal markers. Physical changes to fruit-based products as rheology and colour (expressed as the a*, a*/b*, L or ΔE) are also important markers in shelf-life evaluation (Rocha & Morais, 2003; Ahmed & Ramaswamy, 2005; Oszmiański et al., 2008). Colour changes of fruit products (purées, juices etc.) are often discussed in literature (Ibarz et al., 1999; Chutintrasri & Noomhorn, 2007; Ávila & Silva, 1999). Microbiological shelf-life is not important for fruit baby food because this commodity belongs to non-perishable foodstuffs (Van Boekel, 2009). The aim of the paper is to evaluate shelf-lives of fruit baby food. A procedure for shelf-life determination is shown in the storage experiment bellow.

MATERIALS AND METHODS

Commercially available apple/raspberry fruit baby food from the Czech market was used. Preservation method used: pasteurization; composition: apple puree, water, raspberry puree (20%), sugar, modified corn starch, citric acid, and ascorbic acid; production date: 17th September 2008; best before date: 18th March 2010; lot: 080917.

Shelf-life estimation

Thermal degradation kinetics of fruit baby food filled in original packaging was studied by isothermal heating at selected temperatures (40 °C, 55 °C, 70 °C and 90 °C) for 19 days. The storage test temperatures were chosen according to Rajchl et al., 2010.

DPPH determination

Antioxidant capacity of fruit baby food was determined using the free radical 2,2-diphenyl-1-picrylhydrazil (DPPH) according to (Brand-Williams et al., 1995). Absorbance was measured at 517 nm against blank samples with methanol solution (80%) (Brand-Williams et al., 1995).

Colour measurement

Colour measurements were carried out using the standard CIE L*, a*, b* coordinates on a Minolta CM-2600d spectrophotometer (Minolta, Osaka, Japan). The
measurements were carried out on SCI modality using a 10° standard observer and D65 illuminant. Before the analysis, the instrument was calibrated on a white standard tile \((L^* = 98.82; \ a^* = -0.18; \ b^* = -0.31)\). A colour change was described as:

\[
\Delta E = \sqrt{ (L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (L^* \text{ – lightness, } a^* \text{ – redness/greenness } b^* \text{ – yellowness/blueness. } L_0, a_0, b_0 \text{ indicates initial value of these parameters}).
\]

**Total phenols determination**

Total phenol content was determined using a spectrophotometric assay on a UV-visible Thermo spectronic Genesys 20 spectrophotometer (Thermo Fisher Scientific, Waltham, Massachusetts, USA) and was measured using the Folin-Ciocalteau reaction. (Shaghaghi et al., 2008). Briefly, aliquots of samples and standards (0.5 ml) were mixed with 2.4 mL of deionized water, 2 ml of 2% sodium carbonate (Na\(_2\)CO\(_3\)), and 0.1 ml of the Folin-Ciocalteau reagent. After incubation at room temperature for 60 min, absorbance of the reaction mixture was determined at 750 nm using gallic acid as a standard. Total phenol content was expressed in gallic acid equivalents.

**Ascorbic acid determination**

A homogenised sample (5 g) was weighted into a 50 ml volumetric flask and 0.5% solution of oxalic acid (40 ml) was added. The sample was extracted in an ultrasonic bath at 25 °C for 15 minutes. Then the content was cooled down to 20 °C and filled to volume of 50 ml with 0.5% solution of oxalic acid. The extract was filtered (0.45 µm filter) and ascorbic acid content was analysed. HPLC analyses were performed on a Dionex HPLC (Amedis, Prague, Czech Republic) instrument consisting of a P680 pump, an Ultimate 3000 photodiode array detector and an ASI 100 autosampler controlled by Chromeleon 6.80 software package (Amedis, Prague, Czech Republic). Chromatographic separation of ascorbic acid was carried out by a Phenomex® (Chromservis, Prague, Czech Republic) Synergi 4u Hydro-RP 80A column (4 µm, 250 mm × 4.6 mm i.d.). The column temperature was kept at 30 °C. The injected volume was 20 µl. The mobile phase consisted of 5 mM sulphuric acid in distilled water. Isocratic elution was applied at the flow rate of 1 ml min\(^{-1}\).

**5-HMF and furfural determination**

A homogenised sample (5 g) was weighted into a 50 ml volumetric flask and 10% solution of methanol (40 ml) was added. The sample was extracted in an ultrasonic bath at 25 °C for 15 minutes. Then the content was cooled down to 20 °C and filled to volume with 10% solution of methanol. The extract was filtered (0.45 µm filter) and 5-HMF/furfural content was analysed. HPLC analyses were performed on a Dionex HPLC (Amedis, Prague, Czech Republic) instrument consisting of a P680 pump, an Ultimate 3000 photodiode array detector and an ASI 100 autosampler controlled by Chromeleon 6.80 software package (Amedis, Prague, Czech Republic). Chromatographic separation of 5-HMF and furfural was carried out by a Hibar® RT (Chromservis, Prague, Czech Republic), Purospher® STAR RP-18e column (5 µm, 125 mm × 4.0 mm i.d.). The column temperature was kept at 30 °C. The injected volume was 20 µl. The mobile phase consisted of 10% methanol in water. Isocratic elution was applied at the flow rate of 1 ml min\(^{-1}\).
Sensory analysis
The test room was equipped according to the requirements of the international standard (ISO 8589 – Sensory analysis – General guidance for the design of test rooms). The sensory evaluation was performed by ten panellists (6 female and 4 male) from the Faculty of Food and Biochemical Technology at the Institute of Chemical Technology (PhD students and staff of the Faculty). The assessors were selected, trained and monitored according to the above-mentioned standard (ISO 8586 – Sensory analysis – General guidance for selection, training and monitoring of assessors – selected assessors). Samples from each tested group were served in a session, each time 20 g of each sample in a 50 ml coded beaker. The temperature of all served samples was 20 °C. Samples were neutralized by water and bread. The sample serving was in agreement with the above-mentioned international standard (ISO 6658 – Sensory analysis – Methodology – General guidance). The assessors evaluated differences among the samples using a triangle test (ISO 4120 Sensory analysis – Methodology – Triangle test). Colour, taste and flavour were tested. Two samples of fruit baby food stored at 2 °C were used as a reference material for the triangle test. All analysed samples were from the same lot. The first change of the product was identified by the evaluators when they recognized statistically significant differences between samples (probability level of $P = 0.99$). A consumer preference test was used to determine unacceptability of samples. The samples of fruit baby food were tested by 51 consumers and a product was identified as sensory unacceptable if more than 50% of the evaluators identified it as sensory unacceptable (Lawless & Heymann, 1999).

Data processing
The pseudo first order and zero order kinetic models were used. These kinetic types are expressed by the following equations:

Zero order:

$$c = c_0 + k_0 t$$

(1)

Pseudo first order:

$$c = c_0 e^{k_p t}$$

(2)

where:

$$k_{ps} = k_1 C$$

(3)

where $c$ is the concentration at the time $t$, $c_0$ is the concentration at time zero, $k_0$ is the zero order kinetic constant, $C$ is the concentration of the constant reactant, $k_{ps}$ is the pseudo first order kinetic constant and $k_1$ is the first order kinetic constant. Temperature sensitivity of the rate constant was analysed using the Arrhenius equation:

$$\ln k = \ln k_{ref} - \left( \frac{E_a}{R} \right) \left( \frac{1}{T} - \frac{1}{T_{ref}} \right)$$

(4)

where $k_{ref}$ is the rate constant at the reference temperature ($T_{ref}$), $E_a$ is the activation energy of the studied reaction and $R$ is the universal gas constant.
$Q_{10}$ (temperature acceleration factor) is defined as:

$$Q_{10} = \frac{\text{reaction rate at temperature } (T + 10)}{\text{reaction rate at temperature } (T)}$$  \hspace{1cm} (5)

(Rajchl et al., 2010).

The value of $z$ is the temperature change that causes a 10-fold change in the reaction rate constant.

**Statistical analysis**

The tests were triplicated for each sample and mean values are mentioned. Differences at $p < 0.05$ were considered to be significant. All statistical analyses were performed using Statistica 9.0 (StatSoft CR s.r.o., Prague, Czech Republic) and Excel 2007 (Microsoft Corporation).

**RESULTS AND DISCUSSION**

The following storage experiment shows a general procedure to estimate a shelf-life of fruit baby food. Typical fruit baby food produced from apples and other red-fruit was chosen. Physical and chemical markers potentially suitable for predicting a shelf-life of baby food were selected. Antioxidant capacity, ascorbic acid content and total phenol content were chosen as nutritionally important markers. Colour was chosen as an appropriate parameter for monitoring sensory changes. 5-HMF and 2-furfural contents were monitored, because these compounds are suitable for evaluation of heat treatment of products.

Changes of the selected markers are shown in the plots in Figs 1– 6, and kinetic data ($k$, $E_a$, $z$, $Q_{10}$) calculated from the results of the selected markers are given in Table 1.

![Figure 1](image_url)

**Figure 1.** Changes of DPPH content during the storage of fruit baby-food in various temperatures.
The trend of DPPH changes (see Fig. 1) is ambiguous. The antioxidant capacity decreased during the first 4 days. After the decline, growth followed, which is typical for other types of foodstuffs as well. This trend is probably caused by formation of substances with antioxidant properties during heating, e.g. products of the Maillard reactions, or by increasing availability of substances with antioxidant properties from the food matrix. (Pérez-Conesa et al., 2009; Votavová et al., 2009) Interpretation of changes in antioxidant capacity of food products is generally very difficult because food matrices are very complex and interactions between food components have not been comprehensively described.

The decrease of total phenol content (see Fig. 2) in baby food had exponential trend for the temperatures of 40, 55 and 70 °C. The ‘increase’ of total phenols during heating up to 90 °C was probably due to production of the Maillard reaction products and due to non-specificity of the Folin-Ciocalteu method. The changes in the content of ascorbic acid, furfural and 5-HMF (see Fig. 3 and 4) in baby food had an exponential trend for each selected temperature. 5-HMF and furfural formation at the temperature of 90 °C was not monitored because concentration of those compounds increases dramatically during heating.

**Figure 2.** Changes of total phenols content during the storage of fruit baby-food in various temperatures.
Figure 3. Changes of ascorbic acid content during the storage of fruit baby-food in various temperatures.

Figure 4. Changes of HMF content during the storage of fruit baby-food in various temperatures.

The changes of colour were expressed as $a^*$, $b^*$, $L$ and $\Delta E$ (see Fig. 5 and 6). The $b^*$ parameter decreased at the lower temperatures and increased at the higher temperatures. The changes of the $a^*$ parameter were inconsistent and hardly applicable for data processing.
Figure 5. Changes of colour expressed as $b^*$ during the storage of fruit baby food in various temperatures.

Figure 6. Changes of colour expressed as $\Delta E$ during the storage of fruit baby food in various temperatures.

Colour changes, expressed as $\Delta E$, $a^*$ and $L$, and total phenol contents are applicable for shelf-life evaluation of fruit baby food. Considering that the $z$-value for total phenols is very high, total phenol content is less useful for evaluation of heat treatment or prediction of a shelf-life, but thermal stability of total phenols is advantageous in nutritional terms. Colour is more suitable for shelf-life evaluation, because this marker is one of the most important parameters for consumers. The colour changes correlated well with other markers commonly used for assessment of heat treatment of fruit.
Products. Changes of colour are probably affected by anthocyanin degradation. Ambiguous trends of anthocyanin degradation during heating were observed in literature (Tsai et al., 2005). Colour changes are generally well applicable for assessment of a shelf-life and heat treatment. The calculated kinetic data are given in Table 1. The obtained results roughly correspond with the data of Opatová et al. (1992) and Ibarz et al. (1999). The relatively high standard deviation of the measured data is due to complexity of the matrix and difficulty in evaluating a shelf-life of fruit baby food. The order of reaction for the selected parameters was verified by adjusting the experimental data to the kinetic equations of zero, first and second order using a regression analysis. The changes of total phenols, ascorbic acid, 5-HMF, furfural content and colour parameters expressed as a*, b* followed the pseudo first order kinetics. The colour changes expressed as ΔE and L followed the zero order kinetics (ΔE = ΔE₀ + kt).

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>ka (day⁻¹) (p &lt; 0.90)</th>
<th>Ea* (kJ mol⁻¹) (p &lt; 0.90)</th>
<th>z (°C) (p &lt; 0.90)</th>
<th>Q₁₀ (p &lt; 0.90)</th>
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<tbody>
<tr>
<td>Total phenols</td>
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<tr>
<td>40</td>
<td>28.4 × 10⁻⁶ ± 8.8 × 10⁻⁶</td>
<td>7.2 ± 2.0</td>
<td>245.0 ± 69.0</td>
<td>1.1 ± 0.3</td>
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<tr>
<td>55</td>
<td>32.5 × 10⁻⁶ ± 5.0 × 10⁻⁶</td>
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<tr>
<td>70</td>
<td>36.1 × 10⁻⁶ ± 8.9 × 10⁻⁶</td>
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<td>90</td>
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<tr>
<td>b*</td>
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<tr>
<td>40</td>
<td>6.8 × 10⁻⁴ ± 1.5 × 10⁻⁴</td>
<td>13.5 ± 10.8</td>
<td>127.0 ± 102.0</td>
<td>1.2 ± 1.0</td>
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<tr>
<td>55</td>
<td>8.2 × 10⁻⁴ ± 4.9 × 10⁻⁴</td>
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<tr>
<td>70</td>
<td>10.7 × 10⁻⁴ ± 1.9 × 10⁻⁴</td>
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<td>90</td>
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<td>ΔE</td>
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<td>40</td>
<td>50.5 × 10⁻³ ± 36.7 × 10⁻³</td>
<td>47.0 ± 15.6</td>
<td>43.0 ± 14.0</td>
<td>1.7 ± 0.6</td>
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<tr>
<td>55</td>
<td>101.7 × 10⁻³ ± 82.3 × 10⁻³</td>
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<tr>
<td>70</td>
<td>93.0 × 10⁻³ ± 77.1 × 10⁻³</td>
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<tr>
<td>90</td>
<td>592 × 10⁻³ ± 601.5 × 10⁻³</td>
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<td>L</td>
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<td>40</td>
<td>0.3 × 10⁻³ ± 1.5 × 10⁻³</td>
<td>73.3 ± 81.8</td>
<td>26.0 ± 29.0</td>
<td>2.4 ± 2.7</td>
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<tr>
<td>55</td>
<td>2.1 × 10⁻³ ± 1.9 × 10⁻³</td>
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<tr>
<td>70</td>
<td>1.4 × 10⁻³ ± 2.3 × 10⁻³</td>
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<tr>
<td>90</td>
<td>16.5 × 10⁻³ ± 17.5 × 10⁻³</td>
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<tr>
<td>Ascorbic acid</td>
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<tr>
<td>40</td>
<td>4.9 × 10⁻³ ± 0.6 × 10⁻³</td>
<td>75.4 ± 14.9</td>
<td>29.0 ± 3.2</td>
<td>2.2 ± 0.2</td>
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<tr>
<td>55</td>
<td>21.5 × 10⁻³ ± 2.7 × 10⁻³</td>
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<tr>
<td>70</td>
<td>50.0 × 10⁻³ ± 19.6 × 10⁻³</td>
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<td>90</td>
<td>294.9 × 10⁻³ ± 84.2 × 10⁻³</td>
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<td>5-HMF</td>
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<td>40</td>
<td>29.4 × 10⁻³ ± 15.4 × 10⁻³</td>
<td>46.1 ± 73.4</td>
<td>45.0 ± 72.0</td>
<td>1.7 ± 2.7</td>
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<tr>
<td>55</td>
<td>92.2 × 10⁻³ ± 5.9 × 10⁻³</td>
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<tr>
<td>70</td>
<td>137.0 × 10⁻³ ± 10.0 × 10⁻³</td>
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<tr>
<td>Furfural</td>
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<tr>
<td>40</td>
<td>46.0 × 10⁻³ ± 14.2 × 10⁻³</td>
<td>31.1 ± 26.9</td>
<td>61.0 ± 52.0</td>
<td>1.5 ± 1.3</td>
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<tr>
<td>55</td>
<td>95.4.5 × 10⁻³ ± 31.0 × 10⁻³</td>
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<td>70</td>
<td>143.8 × 10⁻³ ± 20.5 × 10⁻³</td>
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<td>90</td>
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</table>
For shelf-life prediction, changes of ascorbic acid content during storage are not useful due to deliberate addition of this substance to final products. In the end of a shelf-life, baby foods with added vitamin C can contain more ascorbic acid than just produced fruit baby food with no addition of vitamin C. Generally, antioxidant activity is contrary determined by ascorbic acid and therefore, ascorbic acid content was analysed. The results of the accelerated storage experiment suggest definite unsuitability of antioxidant capacity for estimation of shelf-lives.

5-HMF/furfural contents as well as changes are not applicable for shelf-life prediction because formation of these compounds is strongly affected by temperature history of a given product during production (filling, pasteurisation etc.). Changes of carbohydrate content (sucrose inversion) are not a suitable marker in case of baby food because sugar is often added to fruit-based infant food. Colour expressed as b* is not a suitable marker for shelf-life estimation, because the changes of the b* parameter have not an explicit trend.

The example of Arrhenius plots between natural logarithm of $k$ values and $1/T$ is given in Fig. 7; the calculated kinetic equations were used for extrapolation of shelf-lives at the model storage temperatures. The value of sensory difference was determined by the sensory analysis: (unacceptable shift: (L): 37; (∆E): 4).

![Figure 7. Example of Arrhenius plot for total phenols changes.](image)

Figure 7. Example of Arrhenius plot for total phenols changes.

Determination of the first change of a selected marker is statistically more objective, because limits of unacceptability are perceived differently by consumers from different parts of the world. The data were used for construction of shelf-life plots, which are used to estimate storage stability of frozen foods (time-temperature-tolerance) according to Tressler et al. (1968). An example of the shelf-life plots is given in Fig. 8. The shelf-life of fruit baby food calculated according to the L and ∆E at the temperature 20 and 30 °C is given in Table 2.
The shelf-life extrapolated for the average storage temperature of 20 °C corresponds roughly with the shelf-life declared by the producer. Changes of chemical markers, such as total phenols, seem to be generally simpler and more suitable to be used for assessing shelf-lives; however sensorial parameters should be used to determine product unacceptability or the first change of a selected marker. The problem with chemical markers is high variability of their initial concentrations.

CONCLUSIONS

Estimation of shelf-lives is very complicated due to difficulties in determining limits of product acceptability. So far, there has been no definite approach for determination of shelf-lives. Therefore it is necessary to carry out high-quality preliminary research focusing on chemical and physical changes in food commodities before shelf-life testing. Results must be critically assessed and variability of food matrices must be considered. Colour expressed as ΔE and L is the most appropriate marker for shelf-life estimation of fruit baby food. In comparison with chemical markers, colour seems to be a more robust parameter allowing more general estimations. But using colour as a marker for assessing heat treatment and/or a shelf-life is complicated due to thermal damage to input raw material.

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