PROTOCOL

UNDERSTANDING AND IMPLEMENTING ACCELERATED AGEING

Product Requirements & Packaging Optimisation

CITEO x LIBio-ENSAIA

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Introduction to accelerated ageing

Definition and purpose

Accelerated ageing consists of subjecting food to specific conditions to simulate the potential effects of natural ageing, but in a shorter time frame. The process can help predict how a product will deteriorate over time once packaged, without having to observe it over a long period.

It is especially useful at the R&D stage, to test new packaging or evaluate the behaviour of new products. It is also essential for food with a long shelf life, which is sometimes stored under normal conditions over several months or years.

> ⋗ Accelerated ageing is a strategic lever in the eco-design process. It helps optimise the product-packaging pairing while also significantly reducing observation times.

Applications and limitations

✅ Accelerated ageing:

- Is applicable to products with a BBD (best before date) > four weeks
- Is based on physiochemical or sensory tests
- Requires at least two storage temperatures
- Provides reliable results after three to six weeks of storage

❌ Accelerated ageing is not suitable for:

- Products with a UBD (use-by date) < four weeks and concerned by microbiological risks.
- Products that are normally kept at a low temperature (chilled or frozen) and whose state may change in accelerated shelf life conditions (e.g. fusion, glass transition, etc.)

Acceleration factor

The accelerated ageing procedure is based on the calculation of an **acceleration factor** (AF) This factor expresses the speed of ageing in accelerated storage conditions compared with standard conditions. It varies depending on the parameters used and the type of deterioration you want to study (moisture loss, oxidation, browning, etc.).

For example, an "Acceleration factor of 5" means:
Five weeks under normal conditions = one week of ageing under accelerated conditions.

💡 **Please note** – the acceleration factor is relative to the product analysed and its characteristics. Each product has its own acceleration factor.

RECOMMENDATION

- It is better not to exceed an acceleration factor of 10. Beyond that, the speed at which a product deteriorates is no longer realistic and the results will not reflect product ageing in normal storage conditions.

"Step 5: Calculating a product's acceleration factor" describes how to obtain an acceleration factor.

Accelerated ageing protocol

Requirements: Equipment and facilities

To carry out the accelerated ageing procedure, you need an incubator or a controlled temperature and humidity storage chamber.

If the equipment is not available in-house, you can call on the services of an external laboratory.

The procedure set out below can only be carried out under controlled temperature and humidity conditions.

Stage 1: Defining testing objectives

Defining test objectives is essential. They will guide you in your technical choices regarding testing conditions for the following stages of the protocol: analytical tests, duration of tests, ageing parameters, etc.

Simulating how a product changes over time can help you achieve three main objectives:

- **Evaluate the influence of packaging barrier performance on product behaviour**: the idea is to understand how a packaging solution influences product preservation, especially its sensitivity to moisture, oxygen or light.
- **Evaluate and adjust the best before date (BBD)**: it lets you fine-tune product shelf life and avoid a BBD that is too short (risk of wastage) or too long (risk of quality loss).
- **Test a new product formula**: for a change in recipe or a newly developed product, it lets you test product stability, texture, sensitivity to oxygen, etc.

🎯 To optimise your **packaging** so that you meet the exact requirements of your products, the protocol focuses on accelerated ageing used to **study the behaviour of a product in relation to the level of protection provided by the packaging**.

Stage 2: Drawing up the test plan

The test plan will be used to define technical parameters:

- Number of products and packaging to be tested
- Storage conditions
- Sampling frequency (e.g. weekly, monthly, etc.)
- Overall testing period according to the BBD and sampling requirements
- Ageing indicators to monitor, depending on the test, such as:
  - Physiochemical tests (moisture loss, oxidation, texture, etc.)
  - Sensory tests (taste, odour, texture ratings)
  - Microbiological tests

Examples of questions you can ask yourself:

- What type of ageing do I want to study for my product? Which test is best for observing this ageing process?
- How many packaging solutions with different permeability levels do I want to test? How much packaging will I need to package all my products?
- If I carry out sensory tests, how many people will be on the panel – how many products will I need each time sampling takes place?
- How long will the ageing period last (depending on the BBD under normal storage conditions)? How often will I monitor the changes? How many times do I need to take samples?

🍪 CASE STUDY: THE PRODUCT BARRIER REQUIREMENTS PROJECT

With the ENSAIA at the University of Lorraine, we conducted a study on product barrier requirements for the Bread Products, Cakes and Biscuits sector. To do this, we drew up an accelerated ageing protocol as follows (in this example, we refer to one product):

- Number of products tested: 1 cookie product
- Number of packaging solutions with different permeability levels: 3 packaging solutions
- Storage conditions: 2 (normal and accelerated)

Number of different conditions: 1 x 3 x 2 = 6

- Product shelf life: 6 months (product BBD)
- Sampling quantity and frequency: measurement on D0 + 10 more times, spread out over three months of the product's shelf life.
  - Normal at 20°C: t0d, t14d, t28d, t40d, t65d, t90d.
  - Accelerated at 35°C: t7d, t14d, t30d, t45d, t60d.
- Analysis to calculate the acceleration factor: Measurement of product weight gain or loss each time you sample (expressed as a % (g/100g)) → i.e. 3 products measured each time

Number of samples for measuring the variation in weight
= 11 times x 3 samples x 3 packagings = 99 samples

> 💡 **Please note** – other analyses can also be carried out to study several ageing parameters. Don't forget to multiply by the number of samples required for each analysis.

- Additional tests: 5 sensory evaluation sessions with a panel of 15 people

Number of samples for this test
= 5 sessions x 15 people x 6 conditions = 450 samples

Total number of product samples: 99 + 450 = 549 cookies

Stage 3: Defining ageing conditions

Ageing conditions (temperature, humidity, etc.) need to be defined according to the type of product you want to study. Defining ageing conditions involves:

- Having at least 2 temperatures for comparison. Ideally, to calculate the acceleration factor for your product, you should include three temperatures in your test plan, for example: 25°C, 35°C and 45°C.
- It is best not to exceed 45°C, as you run the risk of obtaining erroneous results for your product.

🍪 **CASE STUDY: THE PRODUCT BARRIER REQUIREMENTS PROJECT**

We defined specific conditions to encourage the accelerated ageing of products from the Bread Products, Cakes and Biscuits sector.
Specific temperature and humidity conditions were achieved to create a **double acceleration effect**. The recommended conditions are as follows:

- **Normal conditions**:
  - Temperature: 20°C
  - Relative humidity (RH): 30%

- **Accelerated conditions**:
  - Temperature: 35°C (equivalent to a hot summer in France)
  - Relative humidity (RH):
    - A low RH of 10 to 20% will lead to moist products drying out (retrogradation, crystallisation) ($a_w > 0.5$, e.g. bread, sponge cakes, etc.)
    - A high RH of 50 to 60% will lead to moisture absorption and starch gelatinisation in dry products ($a_w < 0.5$, e.g. cookies, biscuits, etc.)

The a_w (water activity) value is a parameter that indicates the ratio of accessible water in a product.

💡 **Please note** – the conditions presented above are different to the standardised conditions regularly used for packaging characterisation tests (25°C + 75% RH; 38°C + 90% RH; etc.). The goal of the project was to define the right conditions for the product tested.

Stage 4: Applying the accelerated ageing process

Once all the parameters have been determined, you can move on to **applying the accelerated ageing process to products**:

1. Procure products and packaging according to the quantities pre-calculated.
2. Package the products in the various packaging solutions with different barrier properties.
3. Put the products to age in a controlled temperature and humidity storage chamber.
4. Make sure the indicators are monitored over time and take samples as set out in the testing plan for analytical tests or sensory evaluations.

All the data collected will be used to **calculate an acceleration factor** specific to each product or product family with similar characteristics (e.g. “cookies with 5% humidity” product family).

Stage 5: Calculating a product’s acceleration factor

To calculate an acceleration factor that can be used in packaging optimisation, you need to:

- Compare how products deteriorate under the two temperature and humidity conditions chosen:

  In most cases food is found to deteriorate in zero- or first-order reactions.
  - In cases of zero-order reactions, the deterioration is linear.
    See example in Appendix 1 showing a zero-order deterioration and the types of lines produced
  - In cases of first-order (1) reactions, the deterioration is not linear – you need to switch to log data to obtain straight lines.

- Calculate the acceleration factor (AF) according to the following zero-order formula:

  $$\text{FA} = \frac{\text{y T° accelerated}}{\text{y T° normale}}$$

  y corresponds to the gradient of straight lines
  See example in Appendix 1

💡 **Please note** – the acceleration factor includes the notion of activation energy from the Arrhenius model. You can use the Arrhenius model if the humidity is not modified in accelerated shelf life tests, to evaluate the effects of temperature on reaction speed.

Please refer to the Appendix 2

Stage 6: Add accelerated ageing to your range of R&D tools

Once you have calculated the acceleration factor for a given product or product family (with similar characteristics and not modified) and a given set of acceleration conditions (T and RH), you can reuse it and **apply it to all accelerated ageing studies**.

Incorporating accelerated ageing into an eco-design process offers several advantages. It helps:

- **Reduce test times** by switching from several months of ageing to just a few weeks for simulating the compatibility of different packaging solutions with product shelf life goals.
- **Identify the product's barrier requirements precisely** by observing the effects of packaging barrier properties on product preservation to pinpoint the ideal barrier performance required.

  🍪 *Example from the product barrier requirements study – the water vapour barrier requirements for a cookie shelf life of six months is less than* 10 $g.m^{-2}.d^{-1}$
- **Optimise packaging to meet barrier requirements** by comparing test results with material permeability data. You can then choose packaging that is thinner, simpler, more recyclable, etc.

Accelerated ageing can fit in to your packaging optimisation process and is part of an overall environmental impact reduction strategy.

The goal of this protocol is to help you set up R&D projects by simulating the behaviour of product-packaging pairings over a shorter time period: don't hesitate to share it with your teams!

Appendix 1 – Calculating an acceleration factor

Example of calculating the acceleration factor for sliced bread

- Type of deterioration observed: alteration in weight (expressed in %)
- Storage temperatures/humidity: 20°C/30%RH and 35°C/10%RH
- Monitoring period and sampling: 26 days

Product ageing indicator monitored over time at normal and accelerated temperatures, with a zero-order linear reaction (Figures 1 and 2):

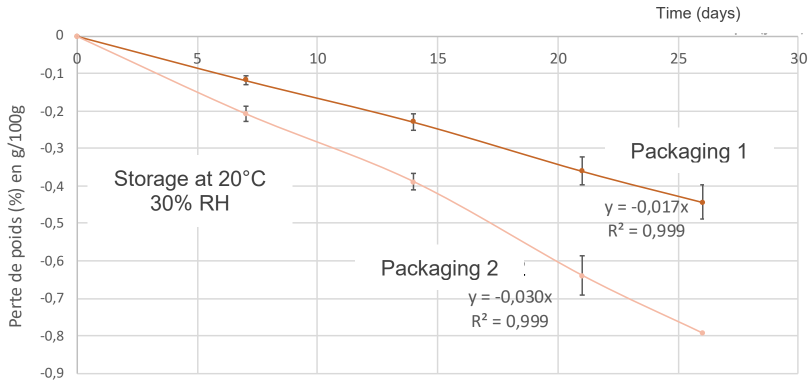

Figure1: Weight loss of the sliced bread in two different packaging solutions (1 and 2), stored under normal conditions: 20°C and 30% relative humidity (RH)

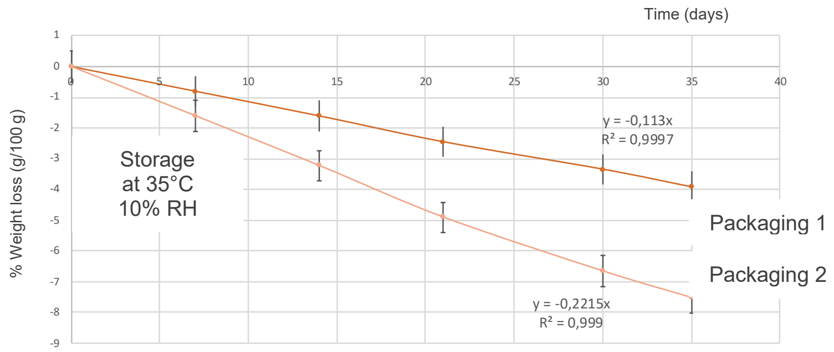

Figure2: Weight loss of the sliced bread in two different packaging solutions (1 and 2), stored under accelerated conditions: 35°C and 10% relative humidity (RH)

A comparison in weight loss between normal and accelerated conditions reveals that after 26 days of storage, the packaged sliced bread samples lost mass in normal (20°C) and accelerated conditions (35°C).

We can observe that more water was lost the when the temperature was higher and the humidity lower.

To calculate the acceleration factor, you need to use the following formula:

$$FA = \frac{y\ T° \text{ accelerated}}{y\ T° \text{ normale}}$$

y corresponds to the gradient of straight lines

The gradient of the product weight loss lines are:

- Product in packaging 1: at 20°C = 0.017%/d, and at 35°C = 0.113%/d
- Product in packaging 2: at 20°C = 0.03%/d, and at 35°C = 0.2215%/d

Under these conditions, sliced bread ageing is accelerated and the acceleration factor is as follows:

$$AF_{\text{Packaging 1}} = 0{,}113/0{,}017 = 6{,}6$$

$$AF_{\text{Packaging 2}} = 0{,}2215/0{,}03 = 7{,}4$$

We can conclude that the average acceleration factor for water loss in sliced bread under accelerated conditions is 7. **This means that a week of storage at 35°C/10%RH corresponds to 7 weeks of storage at 20°C/30%RH.**

The acceleration factor reflects both the higher temperature (faster reactions) and the lower humidity level in the storage chamber (faster dehydration).

Appendix 2 – Arrhenius model

The Arrhenius model is similar to the model for calculating acceleration factors. It is used in cases where only the storage temperature is changed (same humidity level in normal and accelerated storage conditions). This model lets you represent the reaction rate according to temperature.

The Arrhenius model is used to calculate activation energy (Ea).

The product's reaction rate k(T) is calculated by the Arrhenius law as follows:

$$k(T) = k_0 \exp (E_a/RT)$$

Where: T = temperature, k_0 = coefficient, E_a = Activation energy, R = Ideal gas constant = 2 $Cal.K^{-1}.mol^{-1}$ in dry air.

Calculating activation energy based on shelf-life tests involving different temperatures lets you predict the reaction rate at any temperature within the range studied. This model is frequently used in company and laboratory R&D units for the shelf life of products that react to temperature changes and are not very sensitive to ambient humidity.