



## Quality by design for packaging of granola breakfast product

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### ABSTRACT

Quality by Design (QbD) considers both the critical product characteristics and the environmental variables to design an optimum packaging system. This study applied the QbD approach for packaging and shelf-life determination of Granola by i) determining the water vapour transmission rate (WVTR) of packaging films at different environmental conditions, ii) develop and validate a shelf-life model of packed granola breakfast product and iii) predict shelf-life of packed Granola. The WVTR of packaging films (BOPP and biodegradable films, i.e., NK, NM, N913) was measured according to a full factorial experimental design ( $3^2$ ), i.e., 10, 30, 40 °C;  $32.5 \pm 0.5$ ,  $75.5 \pm 0.5$ ,  $92.5 \pm 3.5\%$  RH, and a mathematical model was developed. Granola breakfast product was packed (using the mentioned materials and also a commercial packaging film-control), stored under accelerated conditions (38 °C and 90% RH) and assessed for moisture content (critical quality parameter). A shelf-life model was developed and validated for Granola describing the relationship of the food, packaging and environmental conditions, and shelf-life was inferred for normal storage conditions. The developed WVTR global model considering the dependency of temperature and relative humidity was found to fit the experimental data well ( $R^2 > 0.914$ ). Granola moisture gain was the lowest in BOPP package followed by biodegradable N913 package. The shelf-life for Granola under accelerated conditions ranged from 2 to 13 days depending on the packaging film, and under normal storage conditions (20 °C and 75% RH) was 271, 269, 90, and 33 days for BOPP, N913, NK and NM packages, respectively.

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### 1. Introduction

Granola is a dry granulated cereal product which has a low water activity. Granola's shelf-life is limited by chemical and physical changes and the rate of deteriorative reactions depends on its composition as well as environmental factors. Moisture content was identified as the critical quality parameter and relative humidity as the most influential environmental factor (Macedo, Sousa-Gallagher, & Byrne, 2009). Quantitative determination of quality decay of Granola is important for estimating the shelf-life and designing new processes and packaging (Galic, Scetar, & Kurek, 2011; Macedo, Sousa-Gallagher, Oliveira, Mahajan, & Byrne, 2011).

Strategies often employed to prevent deterioration include control of temperature and water activity, addition of antioxidants, removal of oxygen or modification of headspace gas composition and its retention during distribution and storage, or a combination of these with effective packaging (Kong & Singh, 2011, chap. 2). Packaging represents an essential step of the process, protecting, extending food shelf-life and minimising food waste, while adding

convenience and providing information to consumers. During the distribution chain, Granola can be exposed to a range of quite different environmental conditions, and if there is a gradient between water activity inside and outside the package, this driving force causes a transfer of water molecules through the package leading to an increase of internal water activity, therefore causing an increase of moisture content and consequently a loss of Granola quality.

Environmental concerns have triggered the use of biobased packaging materials as an alternative to materials produced from non-renewable resources. Cereal products are usually packaged in paper/polyethylene packaging (Galic, Curic, & Gabric, 2009), but there is an interest in replacing its packaging and potentially use different and more environmentally friendly packaging materials. NatureFlex manufactures a transparent cellulose base material made from sustainable wood pulp, plus specially formulated biodegradable and compostable surface layers, which control the moisture permeability (<http://www.innoviafilms.com/>).

Moisture transfer in the packaged food depends on the water activity of the food, environmental temperature and humidity conditions of storage, and the permeability of the package to water vapour. Therefore, shelf-life determination is highly dependent on the permeability characteristics of the packaging materials, which emphasizes the importance of packaging design. An optimum

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package design should balance the packaging material properties, product protection requirements, environmental and transport conditions, and cost. Two phenomena should be considered to describe the moisture uptake by a packaged, sensitive food product, (1) the transfer of water vapour through the package, and (2) the kinetic uptake of water by the food product. The shelf-life of a moisture sensitive food product can be estimated using mathematical models that describe and connect the equilibrium sorption isotherm of the product, the initial and the permissible final moisture content, the permeance properties of the package and also the environmental relative humidity and temperature. An experimental evaluation of the optimal packaging design is often avoided due to the restricted time, and predictions using mathematical modelling of quality deterioration (i.e., critical attribute) as a function of factors in the food chain are commonly conducted (Macedo, Sousa-Gallagher, Mahajan, & Byrne, 2011).

There are many references to modelling chemical and physical deterioration and food shelf-life (e.g., Sousa-Gallagher, Mahajan, & Yan, 2011, chap. 2). The most widely studied critical quality attribute is vitamin C in various systems (Feng, Zhana, Qiaob, Wub, & Xiaob, 2012). Shelf-life prediction based on lipid oxidation was used successfully to predict shelf-life of vacuum dried coconut milk powder packed in aluminium foil laminated polyethylene (Jena & Das, 2012). Kulchan, Waraporn, and Suppakul (2010) determined shelf-life of packaged cassava-flour-based baked product, finding good agreements between predicted and experimental findings and demonstrating its application for rapid shelf-life determination. Del Nobile, Buonocore, Limbo, and Fava (2003) predicted the shelf-life of cereal-based packed dry foods, reporting that a constant water permeability coefficient resulted in overestimating the shelf-life of the foods by as much as 90%, therefore modelling of permeability should consider the dependence of water barrier properties on water activity across the film. Lee, Hernandez, Giacin, and Lee (1996) predicted moisture uptake of a moisture sensitive packaged orange chewable multi-vitamins tablet, considering the effect of temperature and relative humidity. Studies of products with months of shelf-life are rare, and Dattatreya, Etzel, and Rankin (2007) is an exception. The most relevant practical gain of modelling shelf-life is to minimise the time required for analysis by securely extrapolating from accelerated to normal conditions. Accelerated shelf-life studies are therefore a well established practice too, e.g., Catauro and Perchonok (2012) target a shelf-life of 4 years.

Incorporating shelf-life models for manufacturing excellence has been recognised by the pharmaceutical industry, which has been very dynamic recently in developing Quality by Design (Waterman, 2011). QbD for packaging and integrative mathematical modelling facilitates simulation making changes of packaging easier and less expensive by reducing the time and experimental work required for its validation.

The aim of this work was to i) determine the water vapour transmission rate of different packaging materials (BOPP, NK, NM and N913) at different environmental conditions, ii) develop and validate an integrative shelf-life model of packed granola breakfast product under accelerated storage conditions, and iii) predict the shelf-life of packed Granola under normal storage conditions. An oral communication was presented at the proceedings of the Seventh International Conference on Predictive Modelling in Foods (Macedo, Sousa-Gallagher, Oliveira et al., 2011).

## 2. Materials and methods

### 2.1. Production of granola breakfast product

Granola breakfast product was produced through wet granulation of cereals (e.g., Oat and Corn flakes, Puffed Rice, Malted

buckwheat, Malted barley) and prebiotic ingredients (i.e., oat beta glucan and inulin) using honey as a binder, in high shear mixers, which involves a size enlargement process with regular shaped granules with a high degree of compaction (Hanley, Pathare, Bas, & Byrne, 2008) and further optimised according to Pathare (2010).

### 2.2. Critical quality parameters

The critical quality parameters were identified by Macedo et al. (2009) by exposing the granola breakfast product to different environmental conditions (temperature and relative humidity). Quality parameters such as moisture content, firmness, colour and particle size distribution were measured throughout storage time and sensory characteristics were also evaluated. Moisture content of granola was measured by the AOAC (1980) oven method at 104 °C for at least 8 h. The texture properties of granola were measured using a TA-XT2i texture analyser, and the firmness ( $N$ ) was expressed as the maximum compression force, the highest peak of the plot force ( $N$ ) versus distance (mm). The colour changes of granola were monitored by the colorimeter Chroma Meter CR-300, using CIELAB  $L$ ,  $a$ ,  $b$  parameters. Sensory characteristics, such as appearance/colour, firmness/texture and overall evaluation/acceptability, were evaluated at different sampling times (0, 3, 8 and 15 day) by a consumer panel using a hedonic scale (1 dislike extremely and 9 like extremely).

The kinetics of food decay are necessary to define a measurable index of deterioration, and analysis is performed by following the variation of each quality index over time during storage, and then comparing the measured value to a threshold. Macedo et al. (2009) found that moisture content MC and firmness could be taken as the indicator of shelf-life and that there is a relative high negative correlation between MC and firmness ( $-0.76$ ), therefore MC was the only factor to be assessed. The Granola critical moisture quality threshold was found to be 8.9% (d.b.), for Granola initial MC of 6.5% (d.b), defined from the correlation between moisture content and the sensory acceptability in terms of texture, for the environmental conditions studied.

### 2.3. Sorption isotherms

The sorption isotherms of dried food products are crucial to model moisture gain during storage and distribution. Macedo, Sousa-Gallagher, and Byrne (2011) determined the Granola isotherms in a range of temperatures found during storage, and an overall model which accounts for the temperature effect on moisture content was developed.

### 2.4. Assessment of water vapour transmission rate (WVTR) of packaging films

The WVTR of the biodegradable packaging films, NatureFlex 30 NK (NK), NatureFlex 23 NM (NM), NatureFlex 55 N913 (N913) and Propafilm RGP 30 (BOPP) was calculated for each film using a full factorial experimental design ( $3^2$ ) at different RH ( $32.5 \pm 0.5$ ,  $75.5 \pm 0.5$ ,  $92.5 \pm 3.5\%$ ) and  $T$  (10, 30, 40 °C). The WVTR of each film was determined by the gravimetric “cup” method described in ASTM E-96-95 (1995). The test cups consisted of a cylindrical cup (6.4 cm bottom diameter, 8.4 cm depth and 7.4 cm top inner diameter) and a lid. In the lid, an opening of 6.6 cm in diameter was made in the centre. The exposed film surface was 43 cm<sup>2</sup>. The cups were filled with a saturated salt solution to provide the equilibrium relative humidity. The different films were placed to cover the opening and secured beneath by the lids, and to guarantee airtight sealing silicon grease was applied around the edge of the cups and under the lids. The cups were kept in dry conditions at different

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