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# An integrated fingerprinting and kinetic approach to accelerated shelf-life testing of chemical changes in thermally treated carrot puree

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ABSTRACT

To have a better understanding of chemical reactions during shelf-life, an integrated analytical and engineering toolbox: "fingerprinting-kinetics" was used. As a case study, a thermally sterilised carrot puree was selected. Sterilised purees were stored at four storage temperatures as a function of time. Fingerprinting enabled selection of volatiles clearly changing during shelf-life. Only these volatiles were identified and studied further. Next, kinetic modelling was performed to investigate the suitability of these volatiles as quality indices (markers) for accelerated shelf-life testing (ASLT). Fingerprinting enabled selection of terpenoids, phenylpropanoids, fatty acid derivatives, Strecker aldehydes and sulphur compounds as volatiles clearly changing during shelf-life. The amount of Strecker aldehydes increased during storage, whereas the rest of the volatiles decreased. Out of the volatiles, based on the applied kinetic modelling, myristicin, α-terpinolene, β-pinene, α-terpineol and octanal were identified as potential markers for ASLT.



## 1. Introduction

Theoretically, when based on microbiological safety, sterilised food can be stored indefinitely. However, the best-before date is limited due to chemical changes whether or not triggered during processing and continuously taking place during shelf-life (van Boekel et al., 2010). Considering consumer expectations, quality should be maintained at a targeted level during the period between processing and purchase as well as between purchase and consumption. There is a need for studies which not only take into account quality changes during processing, but also as a function of shelf-life. For practical reasons, especially when the actual storage time is long, shelf-life studies are based on accelerated shelf-life testing (ASLT) techniques that considerably shorten the process of obtaining the necessary experimental data (Mizrahi, 2000). ASLT works with a basic assumption that the effects of extrinsic parameters (mostly temperature) on the rate of deteriorative reactions can be quantified by applying the principle of chemical kinetics (Labuza & Taoukis, 1990; Mizrahi, 2000; Robertson, 2000). In other words, with the use of increased temperature as abuse condition and assuming that reactions follow Arrhenius kinetics, deterioration rates at ambient/normal distribution conditions can be extrapolated from those of elevated temperatures (Corradini & Peleg, 2007; Hough, Garitta, & Gomez, 2006).

Traditionally, quality investigations during shelf-life have been performed using an univariate approach in which the change in food quality was tailored either with the loss of predetermined quantifiable quality indices such as nutrient or by the formation of an undesirable off-flavour and/or discoloration of compounds (Sithole, McDaniel, & Goddik, 2005). This strategy is the most straightforward way to address such quality investigations, and the corresponding results are undoubtedly of great value. However, fixating on known quality aspects entails that possible different effects are overlooked. Since food degradation is caused by the interaction of many attributes, more comprehensive results can be collected using currently existing food chemometric tools (Saavedra, Cordova, Galvez, Quezada, & Navarro, 2013).

In the present work, to have a better understanding of chemical reactions during shelf-life, advanced analytical methods, relevant data preprocessing methods, multivariate statistical techniques and kinetic models were integrated to develop an analytical and engineering toolbox, called "fingerprinting-kinetics". As a case study, a thermally sterilised carrot puree was selected. Sterilised purees were stored at four different temperatures: 20 °C, 28 °C, 35 °C and 42 °C. In this work, quality changes linked to the volatile

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fraction were studied. Volatiles are often linked to process-induced reactions and have a major contribution to food flavour. Volatiles, being regular degradation products of major food components (e.g., sugar, fat, nutrients), can be approached as indicators for what is happening in a complex food system. The volatile fraction of the samples was analysed with a headspace solid-phase microextraction GC–MS (HS-SPME–GC–MS) fingerprinting procedure as a function of storage times and temperatures (kinetics). This approach considers all compounds detected in the investigated food fraction. Within a fingerprint procedure at the start of the analysis all the compounds are unknowns; it has been called an "untargeted approach" (Grauwet, Vervoort, Colle, Van Loey, & Hendrickx, 2014). The amount of data generated using a HS-SPME–GC–MS analysis might be overwhelming. Multivariate statistical data analysis (MVDA) techniques are most appropriate for extracting important information out of these large data sets by reducing the dimensions of the multivariate data to a few manageable dimensions.

The objective of the present work was twofold. As a first objective, the potential of fingerprinting was used as a fast screening technique for monitoring chemical changes during shelf-life. In other words, fingerprinting could enable selection of volatiles of which detected amounts are changing the most during shelf-life. Only these molecular entities were identified and studied further (to link them to possible reaction pathways). The power of fingerprinting to identify important reaction pathways within a complex of chemical changes was clearly demonstrated in our previous studies (Kebede et al., 2013, 2014a,b; Vervoort et al., 2012).

Since conducting shelf-life studies at normal storage temperatures can be quite resource consuming, as a second objective of this work, the potential of ASLT was investigated. Based on the data from the fingerprinting as a function of storage times and temperatures, kinetic modelling was performed to investigate the reaction kinetics of volatiles (selected by the fingerprinting approach) at different storage temperatures. By evaluating the estimated kinetic parameters, the suitability of these volatiles as quality indices (markers) for ASLT was investigated (Fig. 1).

## 2. Materials and methods

### 2.1. Sample preparation

A single batch of freshly harvested carrots (cv. *Nerac*) was purchased at a local market. The carrots were carefully washed, peeled and cut into standardised cylindrical pieces of approximately 1 cm thickness. The carrot cubes were packed into low-density polyethylene bags. To prevent enzymatic reactions during processing, storage and thawing, the packaged carrots were blanched at 95 °C for 8 min in a water bath (Haake W15 DC-10, Clausthal-Zellerfeld, Germany). The blanching conditions were validated using a qualitative and quantitative peroxidase test (Adebooye, Vijayalakshmi, & Singh, 2008). After blanching, the plastic bags were immediately cooled in ice water for 10 min, frozen in liquid nitrogen and stored in a freezer at −40 °C until processing. Prior to processing, the samples were thawed overnight at 4 °C. In order to prepare the puree, deionised water was added to the blanched carrot, blended for 1 min using a Buchi mixer (B-400; Buchi Labortechnik AG, Flawil, Switzerland) and further homogenised by high pressure homogenisation (at 1000 bar while temperature maintained <4 °C) (Panda 2K, Gea Niro Soavi, Mechelen, Belgium). The sample preparation steps are schematically listed in Fig. 1.

### 2.2. Thermal processing

The thermal treatment was carried out in a static Steriflow pilot retort (Barriquand, Paris, France). An industrially-relevant sterilisation value of $F^{10°C}_{121.1°C}$ ($F_0$) = 5 min was selected. Glass jars (100 mL volume, 95 mm height and 45 mm diameter) were filled with 85 ± 0.5 g of carrot puree and closed with metal lids. Temperature profiles in the retort and at the coldest point of the product were recorded using type T thermocouples (Ellab, Hillerød, Denmark). The data logging device provided real-time information of the whole process. For graphical representation of typical time–temperature profiles of the product and environment during treatment, the reader is referred to Kebede et al. (2013). Following completion of the treatments, samples were transferred to ice water to further cool the product.

### 2.3. Storage

Sterilised glass jars were stored in incubators, protected from light, at 20 °C and 28 °C up to 44 weeks, at 35 °C up to 26 weeks and at 42 °C up to 18 weeks. At fixed points in time (11 points per temperature), glass jars were sampled from the incubators. The vegetable puree was aseptically (next to a Bunsen burner) transferred to small-volume (10 mL) polyethylene terephthalate tubes with polyethylene caps. One gram of sample was taken for

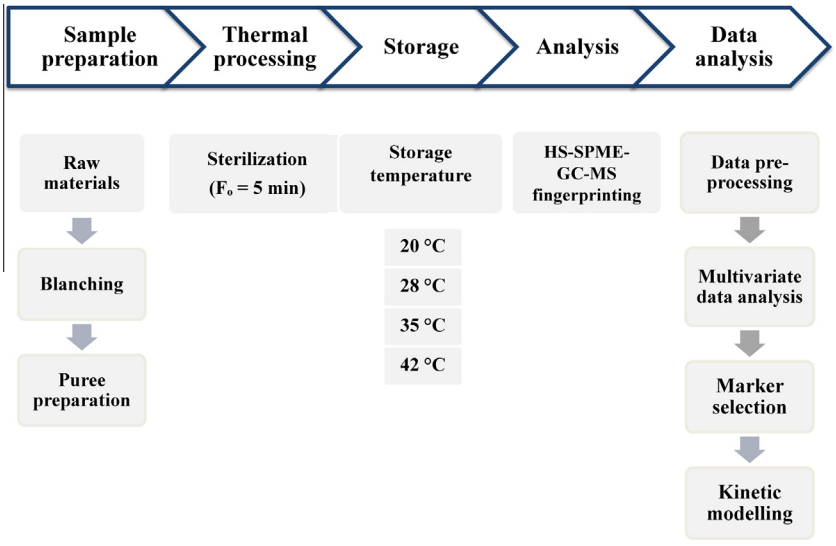


**Fig. 1.** Experimental set-up for investigating chemical reactions in the headspace fraction of thermally treated carrot puree using a fingerprinting-kinetics strategy.