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Shelf life of linseeds and peanuts in relation to roasting

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ABSTRACT

Changes in the oxidative status of peanuts and linseeds during storage were investigated by the ESR spin trapping technique with *N-tert*. butyl-α-phenylnitrone. It has proven to be a suitable method for the determination of the radical generating reactions in the very early stage of fat spoilage and deterioration reactions can still be detected, before changes in sensory properties are noticeable. Primarily, due to oxidative reactions of lipids, shelf life of peanuts and linseeds as well as their sensory quality decreases with storage time. Roasting has a controversial influence on the stability of linseeds and peanuts, respectively. Roasted linseeds became rancid more rapidly than fresh seeds. With increasing roasting temperature and time the oxidative stability of peanuts was improved and shelf life prolonged. This can be attributed to the formation of antioxidant Maillard reaction products. A correlation was found between the amount of deoxyosones as reactive Maillard reaction intermediates and shelf life of roasted nuts.

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1. Introduction

The quality of roasted nuts and oilseeds highly depends on roasting conditions. Besides the formation of desired flavour, by roasting the structure of lipid storage cells was damaged, which eases the oxygen attack. As a result, proceeding of chemical reactions is facilitated, provoking a rapid decrease in the oxidative stability. Lipid oxidation is usually implicated as the most important reason for a decrease in shelf life and generation of undesired flavour. Due to their antioxidant nature, naturally occurring phytochemicals such as tocopherols and polyphenols play an important role in the protection of nuts and oilseeds against fat deterioration. Whereas tocopherols were usually degraded during roasting (Chun, Lee, & Eitenmiller, 2005), e.g. in peanuts the amount of *p*-coumaric acid as predominant phenolic acid with antioxidant potential increased (Talcott, Passeretti, Duncan, & Gorbet, 2005), but changes in polyphenols were poorly related to peanuts' antioxidant properties (Talcott, Duncan, Del Pozo-Insfran, & Gorbet, 2005). Nevertheless, the amount of native antioxidants is only temporarily able to slow down or prevent the lipid oxidation during storage (Talcott, Duncan, et al., 2005). A positive contribution of roasting is the formation of Maillard reaction products, especially melanoidins, which are known to possess pronounced antioxidant properties e.g. (Manzocco, Calligaris, Mastrocola, Nicoli, & Lerici, 2001; Rizzi, 2003) may be able to compensate negative effects and thus improve the oxidative stability of nuts and seeds and prolong rancidity. Using a roasting temperature of 180 °C Hwang, Shue, and Chang (2001) detected a remarkable increase in antioxidant properties of peanuts with increasing roasting time. Also Jeong et al. (2004) found an increasing antioxidant activity with roasting temperature and time for sesame seeds and Abou-Gharbira, Shehata, and Shahidi (2000) detected a positive influence of seeds' roasting on stability of extracted oil. In contrast Talcott, Duncan, et al. (2005) stated that a considerable increase in the rate of fat deterioration in peanuts can be found with increased thermal exposure.

In general, the fat autoxidation process is composed of several radical mechanisms, finally resulting in the formation of compounds as alkylaldehydes and ketones as well as carboxylic acids, mainly responsible for the known rancidity off-flavour. Via commonly used analytical indices as peroxide value (POV) or thiobarbituric acid index (TBARS), reaction products of the advanced stage of fat deterioration are detected. Spectroscopic methods such as fluorescence and infrared spectroscopy proved to complete traditional chemical analyses on peanuts' oxidation status (Jensen, Christensen, & Engelsen, 2004). For prediction of roasted nuts' and seeds' shelf life such methods are not applicable. Instead, reaction products in the very early stage of lipid oxidation have to be determined, appropriate to draw conclusions on the rate of further reactions. A study on chicken meat showed that the concentration of secondary lipid oxidation products such as pentane and hexenal developed similar to the level of radicals formed in the initial stage of fat autoxidation (Nissen, Mannsons, Bertelsen, Huynh-Ba, & Skibsted, 2000). In general the

Abbreviations: OPD, *o*-phenylenediamine; PBN, *N-tert*. butyl-α-phenylnitrone; ESR, electron spin resonance; DH(s), deoxyhexosulose(s).

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steady-state concentration of lipid-derived radicals during fat deterioration is very low and therefore difficult to characterize. The ESR spin trapping technique has proven to be a successful analytical approach for the quantification of such radicals in several foods (Kristensen & Skibsted, 1999; Rohn & Kroh, 2005; Thomsen, Kristensen, & Skibsted, 2000; Uchida & Ono, 1996; Velasco, Andersen, & Skibsted, 2005) and shows a good correlation with results of several conventional detection methods for the oxidative stability. In beer, the so-called "lag time" was considered to be related to the endogenous antioxidant activity (Uchida & Ono, 1996). For chicken meat and beer ESR results also match sensory properties (Nissen et al., 2000; Uchida & Ono, 1996).

The objective of the present study was the development of an ESR spin trapping method applicable for the investigation of nuts' and seeds' oxidative status in a very early stage of deterioration reactions. As a result shelf life prediction via ESR spin trapping was possible. Additionally, the influence of roasting on oxidative stability and shelf life of peanuts and linseeds was investigated. According to the literature an increasing roasting time and temperature result in the formation of Maillard reaction products with antioxidant activity. While linseeds' marginal amounts of free carbohydrates should impede the formation of antioxidant Maillard reaction products during roasting, the oligosaccharide content of peanuts is about 8–10% (Souci, Fachmann, & Kraut, 2008) and an improvement of the oxidative status by roasting was expected. By detection of reactive Maillard reaction intermediates the influence of roasting on lag time and development of sensory properties during storage of peanuts may be estimated.

2. Materials and methods

o-Phenylenediamine (OPD), Fremy's salt and *N-tert*. butyl-α-phenylnitrone (PBN) were purchased from Sigma–Aldrich (Steinheim, Germany), sodium hydroxide and Folin–Ciocalteu reagent were from Merck (Darmstadt, Germany) and gallic acid was from Serva (Heidelberg, Germany).

All reagents were of analytical grade quality with the exception of toluene (Carl-Roth GmbH, Karlsruhe, Germany), which was rotisolv for HPLC.

Fresh linseed and peanut kernels were kindly provided by IGV Institut für Getreideverarbeitung GmbH Nuthetal, Germany.

2.1. Roasting

Roasting took place with a roaster type Novo Plus (Novopak, Germany); roasting conditions were specified by the industry: roasting temperatures for linseed were 120 °C, 140 °C, 160 °C and 180 °C, and 130 °C, 150 °C and 170 °C for peanuts, respectively; roasting times increased from 75 s to 150 s and 225 s (linseed) and 360 s, 480 s, 600 s and 720 s (peanuts), air circulation was between 75% and 95%, filling quantity was 500 g. After cooling (30 min) roasted samples were immediately ground.

2.2. Storage experiments

Prior to storage nuts were ground to obtain homogeneous samples and to accelerate deterioration. Ground samples (1 g) were transferred to 4 mL brown vials and 1 mL of a 7.5 mmol solution of *N-tert*. butyl-α-phenylnitrone (PBN) in toluene was added. The vials were sealed with a rubber septum, and stored in the dark at room temperature (air-conditioned, 22–23 °C) as well as at 35 ± 1 °C in an oven (Heraeus Instruments, Hanau, Germany) for up to three months. Depending on the storage temperature every two to five days samples were taken for ESR-spectroscopy. One milliliter PBN solution was stored under the same conditions and used as blank sample.

Prior to radical measurement 1 mL of a 7.5 mmol PBN solution was added to the stored sample again; it was thoroughly shaken and filtered. This solution was used for ESR measurement without dilution.

2.3. ESR measurement (spin trapping)

Spectra were measured with a Miniscope MS 100 spectrometer (Magnetech, Berlin, Germany). Microwave power and modulation amplitude were set at 10 dB and 1500 mG, respectively, centre field was 3397 G, sweep width 70.7 G. As radical trapping reagent 2 mL of 7.5 mmol PBN solution was used (see above). After storage the obtained solutions (see above) were filled into a 50 μL class capillary (Hirschmann Laborgeräte, Eberstadt, Germany) and measured directly.

All values were performed at least in duplicate and are presented as means with corresponding standard deviation.

2.4. Evaluation of ESR results

The peak area of the second line in the EPR spectrum of the PBN radical adduct is used as a measure for the amount of radicals trapped. This value is calculated for 1 g nut and explained as relative radical intensity. Determination of PBN radical adduct concentration was by external calibration, with Fremy's salt in toluene as standard, based on the method described by Velasco et al. (2005). One millimole of Fremy's salt corresponds to a relative radical intensity of 20 560.

Lag time is defined as the induction time for the formation of radicals as a result of deterioration reactions and used as a measure for oxidative stability (Uchida & Ono, 1996). It was calculated via the intercept point of the horizontal tangent (zero up to the relative radical intensity of 1000) and the tangent which starts with the slope in the curve (relative radical intensity around 1000).

2.5. Detection of the total phenolic content by Folin–Ciocalteu method

Gallic acid equivalents (GAE) were determined according to the method of Singelton, Orthofer, and Lamucia-Raventos (1999). 2.5 g of the ground samples were suspended in 10 mL of distilled water and allowed to stand overnight. The samples were centrifuged at 6000 rpm and 250 μL of the clear solutions were used for detection. The calibration curve was set up by using gallic acid in the concentration range between 5 and 100 mg/L.

2.6. Quinoxalines

2.6.1. Preparation of quinoxalines

After roasting the nut samples were allowed to cool down for 30 min and ground. Five grams of the samples were instantly treated with 10 mL of a 0.05 mol aqueous solution of *o*-phenylenediamine (OPD). After 30 min reaction at room temperature the samples were filtered and the solution was used for analytical investigation without isolation of the quinoxalines formed.

2.6.2. Analytical detection of quinoxalines

α-Dicarbonyls were qualitatively and quantitatively detected as quinoxalines by HPLC and after extraction with *n*-butanol by GC–MS according to the literature (Fiedler, Moritz, & Kroh, 2006; Hollnagel & Kroh, 2000). All values were obtained at least in duplicate and presented as means with corresponding standard deviations.

2.7. Sensory investigations

For sensory investigations about 200 g of ground samples from the same batches as used for PBN investigations were put into glass flasks

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