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Investigation of the roasting, pressing and storage process of highly unsaturated oils

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AFFIDAVIT

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Abstract

Cold pressed oils are often rich in highly unsaturated fatty acids and through this technique other valuable ingredients are kept in the oil. Flaxseed, chia seed and hempseed oil from the oil mill Fandler are produced using a cold pressing technique and contain a high concentration of unsaturated fatty acids. The flaxseed oil is especially sensitive to oxidation and it is therefore advised to store it in a cool place. The chia seed and hempseed oil are not as sensible to oxidation and can therefore be stored at room temperature.

The aim of this thesis was, on the one hand, to characterise the three oils and, detect crucial processing parameters during the roasting, pressing, storage and filling process. To the best of the authors knowledge the roasting, pressing, storage and filling process of either chia seed, flaxseed or hempseed oil has not been studied so far. On the other hand, a rapid test method was validated, as it is in use at the oil mill Fandler to provide a rapid test method for chosen fat classification values. These values are determined mainly to give an idea of the oxidation state of a certain oil.

To validate this method, the fat classification numbers, according to current regulations were obtained parallel to the determinations using the rapid test method. Samples of the roasting and pressing process of all three oils were taken at the oil mill Fandler and investigated at the Institute of Analytical Chemistry and Food Chemistry. To determine the volatile compounds of theses samples as well as from bottled oils with different best before dates, Headspace Solid Phase – Microextraction (SPME) Gas Chromatography – Mass Spectrometry (GC-MS) was used and Odour Activity Values (OAV) of the roasting process were calculated. With the sensory expert panel of Graz University of Technology sensory evaluations of the roasting and storage process of hempseed oil, of all three bottled oils as well as evaluations of sniffing sticks were performed.

In the first part of this thesis, measurements done with the rapid test method used at oil mill Fandler were compared to measurements done for these fat classifications value using standardized techniques. Great differences could be found between the rapid test method evaluations and the determination using standardized techniques. As this was the minor part of the study at hand and could not be further investigated with laboratory equipment, the data was handed over to the provider of the rapid test method and will be tackled by the provider himself.

In the second, and main part of this thesis, primary the volatile compounds of chia seed, flaxseed and hempseed and their according oils were investigated during the roasting, pressing and storage process. The roasting process showed that measured surface temperatures for the oils were rather different and the highest temperatures were obtained, as expected, for the hempseeds. These temperatures are needed to build the typical aroma forming volatile compounds in the seeds. In the hempseed oil pyrazines were determined and found to lead to a roasty and nutty aroma. Overall it was found that most of the flavour compounds of the investigated oils are gained either over the course of roasting or can also develop while they are stored. All investigated oils showed primary oxidation products and occasionally occurring secondary oxidation products. It could be shown that the oxidation products form mostly over the period of storage in bigger tanks. The comparison of oils with different best before dates (BBD) at different stages of their storage showed an overall decrease of aroma forming volatile compounds in all three oils. As expected, the oxidation products increase with prolonged storage over the BBD. The fatty acid composition of all three oils was determined as well for all three oils and showed the expected elevated levels of linolenic acid for chia seed and flax seed oil, and higher levels of linoleic acid for hemp seed oil. Despite these results, the detailed formation of flavour and oxidation compounds is still not resolved fully and needs further investigation.

Kurzfassung

Kalt gepresste Öle sind meist reich an hochungesättigten Fettsäuren, die diese Öle meist weniger oxidationsstabil im Vergleich zu anderen Ölen machen. Wird die Technik einer Kaltpressung verwendet, werden auch andere wertvolle Stoffe, wie z.B. ω-3-Fettsäuren erhalten. Ziel dieser Arbeit war es einerseits, die ausgewählten Öle, Chiaöl, Leinöl und Hanföl zu charakterisieren und entscheidende Prozessparameter zu erkennen und die Veränderungen der Samen über den Röstprozess und Veränderungen im Öl während dem Pressen, Lagern und Abfüllen zu untersuchen. Nach dem Kenntnisstand der Autoren wurde der Röst- und Lagerprozess weder von Chiaöl noch Leinöl noch Hanföl bisher untersucht oder dokumentiert. Andererseits wurde eine Schnelltestmethode zur Bestimmung der Fettkennzahlen, die bei der Ölmühle Fandler verwendet wird, im Vergleich zur klassischen Bestimmung dieser Fettkennzahlen validiert. Proben des Röstens und Pressens aller drei Öle wurden in der Ölmühle Fandler entnommen und am Institut für Analytische Chemie und Lebensmittelchemie untersucht. Diese Proben und Proben von bereits abgefüllten Ölen mit verschiedenen Mindesthaltbarkeitsdaten, wurden mittels Headspace SPME GC-MS untersucht. Diesen Messungen wurde ein interner Standard zugesetzt der anschließend die Berechnung von Geruchsaktiviätswerten (OAV) möglich machte. Zusätzlich wurde eine sensorische Evaluierung der Hanfsamen und des Hanföls, sowie der drei bereits gefüllten Ölen durchgeführt, weiters wurden auch Riechstreifen Verkostungen mit dem sensorischen Expertenpanel der Technischen Universität Graz abgehalten.
Im ersten Teil dieser Arbeit wurden Messungen, die mit der Schnelltestmethode bei der Ölmühle Fandler durchgeführt wurden, mit Messungen verglichen, die für diese Fettklassifizierungswerte unter Verwendung standardisierter Techniken durchgeführt wurden. Große Unterschiede konnten zwischen den Auswertungen des Schnelltestverfahrens und der Bestimmung mit standardisierten Techniken gefunden werden. Da diese Validierung zusätzlich zum Hauptteil der Arbeit durchgeführt wurde und die notwendigen Untersuchungen nicht weiter möglich waren, wurden die ermittelten Daten an den Anbieter der Schnelltestmethode übergeben und werden vom Anbieter selbst evaluiert. Im zweiten Teil, und Hauptteil, dieser Arbeit wurden primär die flüchtigen Verbindungen von Chiasamen, Leinsamen und Hanfsamen sowie deren entsprechenden Öle im Röst -, Press - und Lagerprozess untersucht. Der Röstprozess zeigte unterschiedliche gemessenen Oberflächentemperaturen für die untersuchten Öle, die höchsten Temperaturen wurden, wie erwartet, bei der Röstung der Hanfsamen gemessen. Diese Temperaturen werden benötigt, um die typischen aromabildenden flüchtigen Verbindungen in den Samen zu bilden. Im Hanföl wurden Pyrazine festgestellt, die zu einem röstigen und nussigen Aroma führen. Insgesamt wurde festgestellt, dass die meisten aromabildenden flüchtigen Verbindungen der untersuchten Öle entweder während des Röstens gewonnen werden aber sich auch während der Lagerung entwickeln können. In allen untersuchten Ölen zeigten sich primäre Oxidationsprodukte und gelegentlich auftretende sekundäre Oxidationsprodukte. Es konnte gezeigt werden, dass die Oxidationsprodukte meist über die Lagerzeit in größeren Tanks entstehen. Der Vergleich von Ölen mit verschiedenen Mindesthaltbarkeitsdaten in verschiedenen Phasen ihrer Lagerung zeigte eine Gesamtabnahme von aromabildenden flüchtigen Verbindungen in allen drei Ölen. Wie erwartet steigen die Oxidationsprodukte bei längerer Lagerung über das Mindesthaltbarkeitsdatum. Die Fettsäurezusammensetzung wurde ebenfalls für alle drei Öle bestimmt und zeigte die erwarteten erhöhten Werte von Linolensäure für Chiasamen und Leinsamenöl und höhere Konzentrationen von Linolsäure für Hanföl. Die detaillierte Bildung von aromatischen und oxidativen Verbindungen ist aber immer noch nicht vollständig geklärt und bedarf weiterer Untersuchungen.

Table of content

Abbreviations

ALA	α-Linolenic Acid
BBD	Best Before Date
EI	Electron Ionization
FAME	Fatty Acid Methyl Ester
FID	Flame Ionisation Detector
GC	Gas Chromatography
GC–FID	Gas Chromatography–Flame Ionisation Detector
GC-MS	Gas Chromatography–Mass Spectrometry
GLA	γ-Linolenic Acid
LOD	Limit of Detection
LOQ	Limit of Quanitification
MeOH	Methanol
MS	Mass Spectrometry
NaOH	Natriumhydroxide
OAV	Odor Acitivity Value
PCA	Prinicipal Component Analysis
QDA	Quantitative Descriptive Analysis
RI	Retention Index
SPME	Solid Phase Micro Extraction
THC	Tetrahydrocannabinol

1. Introduction

The aim of this study was to gain detailed knowledge of the behaviour of selected oils of the oil mill Fandler over the whole production and storage process. The oil mill Fandler produces high-quality vegetable-based oils, which possess a nutritionally high-quality composition of fatty acids as well as product-specific sensory properties. To extract the oil from the oilseed, it is necessary to squeeze the oil seed to break up the seed hulls so that the oil is made available for the pressing process. Furthermore, the squeezed oilseed is warmed slightly to increase the oil yield. For some oils - such as pumpkin seed oil or hempseed oil - the oilseed is additionally roasted to form the product-typical nutty notes in the product. In this case, temperatures of higher than 100°C in the roasting pan are necessary for the needed reactions to take place (e.g. Maillard reaction and Strecker reaction). However, the high proportion of polyunsaturated fatty acids in the investigated edible oils make these products easily prone to oxidative processes. The formation of volatile oxidation products (mostly odour active carbonyl compounds) is characteristic in such products and cannot be avoided during the manufacturing and storage process. In low concentrations, these compounds are desirable because they contribute to the formation of the product-typical flavour. However, as the oxidation progresses, and the odour-active oxidation products appear in higher concentrations, the odour is perceived as rancid and has a negative effect on the aroma. For best product quality, it is not only necessary to work with selected raw materials of high quality, but also to know the chemical changes of the oil throughout the production and storage process. For this reason, the production process of selected oils starting from the oilseed to the finished product was closely analysed for the purpose of the present master thesis.

In addition, the knowledge and determination of basic fat indices as well as the fatty acid composition of the oils is important to characterise the products. The evaluation of a quick test to determine the fat indices was therefore another focus of this study.

In the theoretical part of the thesis at hand the background considering the production and properties of cold pressed oils and details to the investigated oils are found. Also, legal guidelines and a chemical background for the non-enzymatical browning and lipid oxidation is given. In the next part the used methods and a detailed description of those can be found. After the used methods and their theoretical background, the experimental part follows. In this part the material and used methods can found in more detail and with all used settings and concentrations. Following the results and discussion is given. All results gathered in this thesis are compared with literature and discussed further on. In the last chapter a final conclusion will be given. After the conclusion the appendix with additional tables, as well as the references and the list of tables and figures can be found.

2. Background

2.1. Flavour compounds

If food is consumed by humans, taste-, odour- and touch-sensations are combined under the word "flavour". The compounds that are responsible to cause the flavour sensation can be categorized in taste and aroma compounds. Taste compounds are mostly not volatile at room temperature and are only recognized by the taste receptors. Considering taste compounds, sour, sweet, bitter, salty and umami are known sensations. In the last years, also the taste sensation of fat has been discovered. As humans and mammals are often drawn to food rich in lipids, a taste ability devoted to the perception of lipids was investigated. This taste sensation can also be called an oleogustus taste sensation [1]. Another taste sensation that has been under investigation in the last years is the taste of kokumi, which enhances the taste of salty, sweet and umami tastes. Kokumi is found as a taste enhancer and not having a taste itself [2]. The taste sensations described as thick flavour or mouthfulness are also provoked and enhanced by kokumi compounds [3]. In contrast, the aroma compounds, are volatile compounds that can be detected by the smell receptors of the human body. These receptors are reached by the air inhaled through the nose (orthonasal perception) or via the pharynx after the chewing (retronasal perception) as seen from Figure 1.

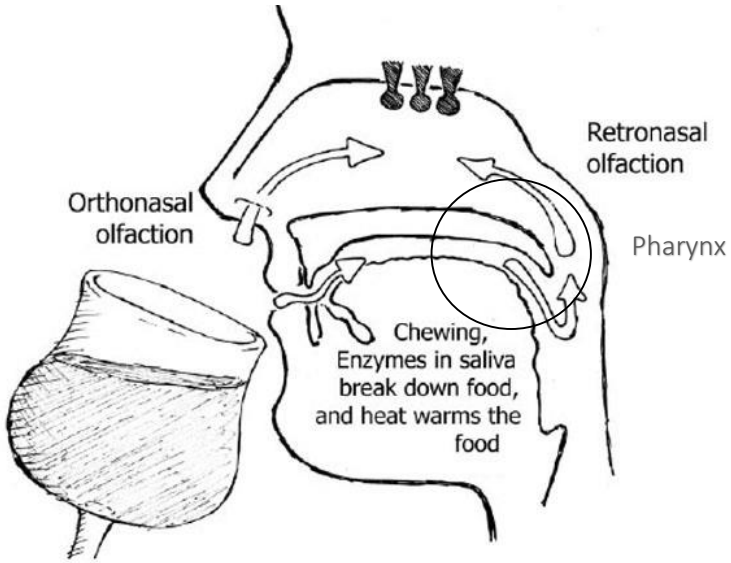


Figure 1: Orthonasal and retronasal perception/olfaction [1]

Considering aroma compounds, especially compounds that give the characteristic aroma to a certain food are termed as character impact compounds. Latest research has shown that from approx. 10 000 identified volatile compounds in food, only around 250 compounds are found to be character impact compounds [4]. The concentration of a compound that is just enough to recognize it by its smell is called the odour threshold (or recognition threshold). The detection threshold is lower than the odour threshold and is described as the concentration at which the compound is recognizable, but the odour cannot be defined precisely. In most foods the odour threshold value is between mg/kg to µg/kg, sometimes even lower. These recognition thresholds are defined using smelling (orthonasal value) or tasting the sample (retronasal value) in different dilutions. But often only orthonasal values are determined.

[1] Adapted from http://drinks.seriouseats.com/images/2013/06/Ice_orthonasal.jpg

Using these recognitions thresholds, the impact of flavour compounds on the overall flavour of investigated food can be estimated and judged. Using the concentrations of the determined thresholds, the so-called odour activity value (OAV) can be calculated if the concentration of a compound and the respective odour threshold is known. For these calculations Equation 1 is used. The flavour value concept was first implanted by Rothe and Thomas in 1963 and recognizes the concentration of a compound in the food matrix besides the odour threshold.

$$OAV = \frac{c_x}{a_x} \qquad \textbf{Equation 1}$$

c_x	Concentration of compound x in the investigated food
a_x	Odour threshold of compound x in the investigated food

However, only compounds with an OAV ≥ 1 can have an impact on the overall formation of the characteristic aroma profile. The importance of a certain compound for the formation of the characteristic aroma is increased accordingly to an increased OAV. The concept of the OAV considers, that certain compounds can have synergetic effects on the aroma of one another.
The biggest limitation of the OAV concept is the impact of the sensory determination method of threshold values as well as the dependence on the matrices the values have been determined in [4]. Until today it is hardly possible to predict the flavour of a certain compound based on its chemical structure. Interestingly, it has been found that the geometrical structuring of a compound can influence the flavour impression. In most foods the odour or flavour is derived from the interaction of a variety of aroma compounds. Over 200 different aroma compounds have been identified in some foods. Dunkel *et al.* [5] reported in 2014 that mostly 3-40 compounds in a characteristic ratio form the typical odour of a certain food. Another important factor, as already mentioned, is the concentration of a certain compound, as this can influence the aroma impression additionally. For example, α -ionon has a cedar wood like odour impression, but if it is diluted with alcohol the odour impression changes in a violet type of odour. Flavour formation can be derived from a variety of pathways. Aldehydes and ketones are obtained mainly from lipids through different metabolic pathways like hydroxacid cleavage, beta oxidation or lipoxygenase catalysed oxidation. Additionally, acids, alcohols, lactones and esters can be formed through oxidations, reductions and esterification. The deamination of amino acids which is followed by decarboxylation can lead as well to volatile compounds. Volatile compounds such as aliphatic and branched chain alcohols, acids, carbonyls and esters are formed [6].
As already mentioned briefly above the perception of off-flavour is also possible. The term off-flavour is used to describe foreign, usually not occurring flavour in the investigated food, that can arise if key aroma compounds are getting lost or the concentrations of certain aroma compounds increase. [7]. The formation of these off-flavours is illustrated in Figure 2. Off-flavour formation can have different causes, one possibility are chemicals that are transmitted via air, water or packaging onto the food, the main problematic chemical compounds are chlorophenols or chloroanisols. Infestation can lead to off-flavour in food, as for example, algae can release compounds like geosmin and 2-methyl-i-borneol which lead to earthy odour. Also, numerous off-flavours are formed by chemical altering of food ingredients, such as the influence of oxygen on certain terpenes or lipid oxidation [5].

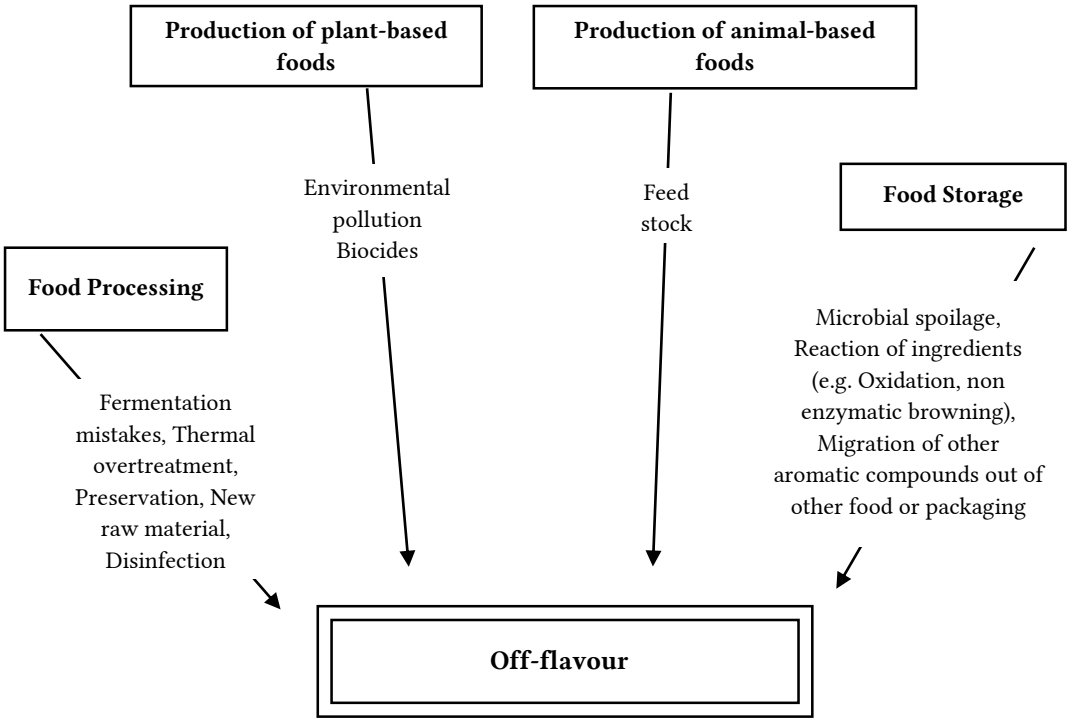


Figure 2: Formation of off-flavour [2]

2.2. Cold pressed oils

Cold pressed oils have the advantage that only gentle thermal treatment is applied during the roasting process and not while pressing, afterwards no refining is applied or needed. Based on these mild processing parameters, the typical sensory properties are transferred into the final oil. Additionally, all accompanying substances – that are in many cases considered health beneficial - are maintained in the oil. Often it is not necessary to add any antioxidants as the natural antioxidants in the oil are preserved. The taste and smell of the produced oils is usually typical for the processed seeds and can vary, for example, depending on the cultivation area, the harvest time, climate and variety. In general, edible fats and oils contain mixtures of mono-, di- and tri-esters of the trivalent alcohol glycerol with different, mostly even-numbered and unbranched aliphatic monocarboxylic acids. This kind of acids are also known as fatty acids. The different fats we know are distinguished because of their consistency. The melting point of a fat is linked to the fatty acid profile of the oil or fat. It is furthermore possible to distinguish between oils which are fluid at room temperature in drying oils like flaxseed or poppy seed, semi drying oils like peanut or rape seed oil and not drying oils like olive oil. These classifications are used to categorize the different tendency for autocatalytic oxidation in the presence of oxygen and are based on the amount of polyunsaturated fatty acids in particular [8].

The higher the amount of the polyunsaturated fatty acids the faster the oil will “dry” (resinous) under atmospheric conditions [9]. The cold pressed oils that are produced at oil mill Fandler and are evaluated in this thesis are pressed using a compactor press, as seen from Figure 3. These can be summarized with the extruding press under the topic of closed continuous squeezers. Most of the produced oils are made using a compactor press.

[2] Adpated from Belitz, Textbook of food chemistry

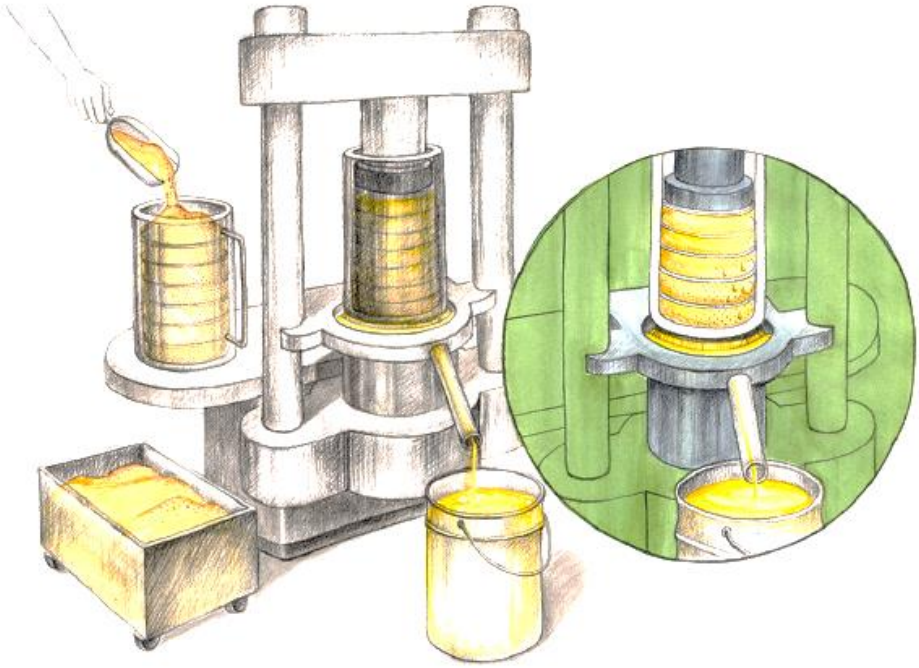


Figure 3: Operating mode of a compactor press [3]

In the first step, little seeds like flax or chia seeds are squished prior to warming. The oil seeds are then carefully warmed in small batches. After warming of the oil seeds to approx. 100°C, depending on the oil seed, the seeds are transferred into the press and layered with pressing disks. Gentle pressure is applied onto the seeds to extract the oil [10]. The pressure is increased gradually starting, for instance, for chia seed oil at 100bar for 30 sec., increasing to 200 bar for 90 sec., 300 bar for 10 sec., 400 bar for 10 sec. until 590 bar are reached and kept for 2000 sec. Advantages of cold pressed oils are the preservation of the natural and precious accompanying substances, such as waxes, enzymes, β-carotin, lecithin, vitamin E and other secondary plant substances of the oil. Additionally, no organic solvent is used and no antioxidants are needed, as the oils still have a great amount of their natural antioxidants. As mentioned above, especially the polyunsaturated fatty acids are preserved through cold pressing of the oils, as illustrated in Figure 4. A disadvantage of cold pressed oils is the possible presence of undesired residues (e.g., pesticides, heavy metals or polycyclic hydrocarbons) and fat degradation products that would be removed during a subsequent refining process. This is the reason why certain limits of pesticides or heavy metals in the raw seeds should not be exceeded. Because of their usually high content of unsaturated fatty acids those oils are also not heat stable, for instance, if linolenic acid is heated for a longer time, decomposition products such as HNEs (4-hydroxy-trans-2-nonenale) are formed. Because they contain higher amounts of polyunsaturated fatty acids, the shelf life of these oils is shortened as well [8].

[3] https://www.fandler.at/cms/wp-content/uploads/2016/10/oel_03.jpg

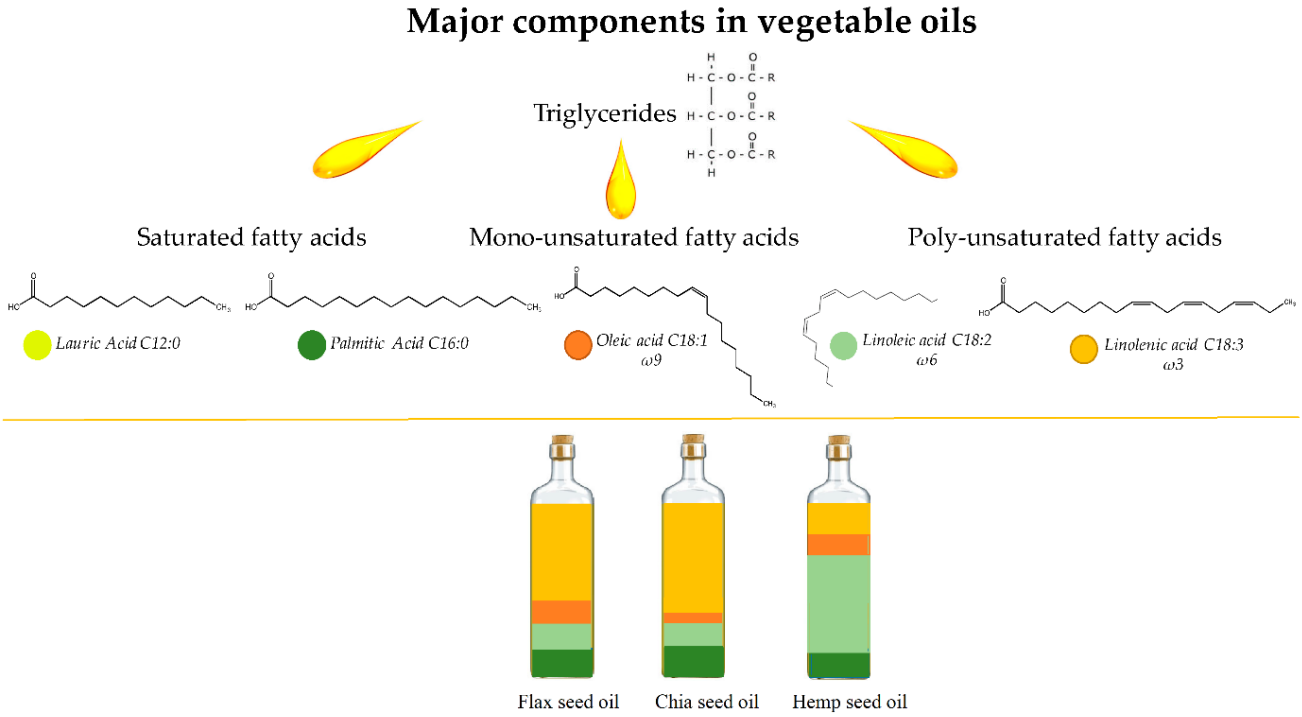


Figure 4: Major components in vegetables oils and composition of the investigated oils [4]

2.3. **Chia seed oil**

Chia seeds are harvested from *Salvia hispanica*, shown in Figure 5 which is a plant indigenous to South America. As the plant is very intolerant to cold climates, it is typically grown in greenhouses throughout Europe and Latin America. Usually, the seeds of the chia plant are white or black coloured and show an oval shape. The colour of the oil is described as light yellow, but the colour can vary depending on the pigment concentration of the seeds themselves (Figure 6). In chia seed oil carotenoids have been found, but no chlorophyll pigments. The odour of the oil is described as not recognizable to lightly nutty; the flavour has been found to be neutral [11].

[4] Adpated from: http://www.mdpi.com/molecules/molecules-22 01474/article_deploy/html/images/molecules-22-01474-g001.png

Figure 5: *Salvia hispanica L.* plant [5]

Figure 6: Chia seed oil and chia seeds

2.3.1. Fatty acid composition

Chia seed oil is rich in polyunsaturated fatty acids; an average composition of chia seed fatty acids is given in Table 1. The ratio of polyunsaturated fatty acids and saturated fatty acids is reported to be 1:7.9, whereas the ratio of ω-6- to ω-3-values is 1:0.29 [12].

Table 1: Fatty acid composition of chia seed oil (Source: [8])

	Fatty acids	Content [g/kg]
C16	Palmitic acid	71
C18	Stearic acid	33
C18:1	Oleic acid	60
C18:2	Linoleic acid	188
C18:3	Linolenic acid	641

2.4. Flaxseed oil

Flaxseeds, or sometimes called linen seeds, are harvested from *Linum usitatissimum L.,* as shown in Figure 7, and are one of the oldest crops in the world. Nowadays, flaxseed is planted all over the planet, but the biggest producer of flaxseeds worldwide by far is Canada, followed by Russia and China. The seeds of the plant are rich yellow to dark brown, the shell of the seed is very hard, and the shell contains oil and carbohydrates very sparingly. Flaxseed can be grown in nearly every climate, but if the climate is very hot and dry the oil content of the seeds is reduced.

[5] https://www.exklusive-chiasamen.de/wp-content/uploads/bluehende-chia-pflanze-e1439660414219.jpg

Flaxseed oil can be won if the seeds are pressed cold after they were hackled. If the oil should be used for technical applications, it is extracted with solvents and then refined. The oil itself has a golden to yellow colour if it is cold pressed, when it is extracted with solvents it shows a lighter colour (Figure 8). The odour of the oil is very spicy, and the taste of the oil is reported to be a bit nutty and sometimes reminding of hay [8]. The seeds of the crop are coated with mucilage which makes it sticky if it is wet. The hull of the seed makes up for approx. 20 % of the seed. The oilseed contains approx. 38-45 % of oil, 28 % dietary fibre and 4 % ash. But significant differences in the composition of flaxseeds have been reported from different cultivars. Albumin and globulins are the major proteins in flaxseeds [13]. The overall protein content of flaxseeds varies from 20-30 % [14]. In seed processing, the water content is very crucial. If the moisture content is increased from 8 % to 16 % the oil yield is reduced from 54,7 % to 4,4 %. Dedio *et al.* [15] suggested that the loss in oil yield is depending on the mucilage development in the outer epidermal cells of the seeds. As water is added the mucilage swells, which reduces the rupturing of the seeds and the internal tissue that contains the oil [15]. If flaxseed oil is used for frying (177-190°C), a fishy flavour can be recognized possibly caused by 1-penten-3-one. The oil oxidation products that are formed can be different from one another at low and high temperatures [16]. Also, the formation of 5- and 6-membered ring cyclic fatty acid monomers from α-linolenic acid (ALA) has been reported. The heating of ALA can also cause the formation of trans-isomer and toxic furan fatty acids such as 2-pentyl furan [17].

Figure 7: *Linum usitatissimum L.* plant [6]

Figure 8: Flaxseed oil and flaxseeds

2.4.1. Fatty acid composition

Flaxseed oil is rich in polyunsaturated fatty acids; an average composition of flaxseed fatty acids is given in Table 2. Flaxseed oil contains 10-12 % glyceride of saturated fatty acids such as oleic acid, linoleic acid amounts to 16-25 % and linolenic acid amounts to 40-62 % [18].

[6] https://canna-pet.com/wp-content/uploads/2015/04/flax-info0.gif

Table 2: Fatty acid composition of flaxseed oil (Source: [19])

	Fatty acid	Weight percentage applied to total fatty acids
C16	Palmitic acid	4,0-6,0
C16:1	Palmitoleic acid	NN-0,5
C18	Stearic acid	2,0-3,0
C18:1	Oleic acid	10,0-22,0
C18:2	Linoleic acid	12,0-18,0
C18:3	Linolenic acid	56,0-71,0
C20	Arachidic acid	NN-0,5
C20:1	Eicosanoic acid	NN-0,6

2.4.2. Volatile compounds

Flaxseed oil has a very characteristic smell and taste; volatile compounds contribute to the typical sensory impression. Over 60 volatile compounds have been identified in flaxseed oil [20]. The identified compounds were categorized into 8 groups: 20 aldehydes, 7 ketones, 13 alcohols, 8 carboxylic acids, 3 esters, 3 alkanes, 4 heterocyclic compounds and 2 other compounds. 53 aroma active compounds could be identified in the oil. The ones with the highest intensity were (*E, E*)-2,4-pentadienal (green, oily), (*E,E*)-2,4-heptadienal (sweet, hazelnut, woody), 5-ethyldihydro-2(3H)-furanone (cereal like), 1-hexanol (herbaceous, woody, green), acetic acid (sour, pungent, strong) and γ-butyrolacetone (sweet, caramel). Most of the detected compound species originate from the oxidation of linolenic compounds, which dominate the composition of linseed oil, while 2-propenal, pentanal, hexanal, 2,4-decadienal, and hexanoic acid, among others, were released from linoleic compounds. The concentration of hexanal (7–26 ppm), *(E)*-2-pentenal (25–39 ppm), 1-penten-3-ol (3.1–4.8 ppm), (*E,E*)-2,4-heptadienal (33–50 ppm), (*E,E*)-2,4-decadienal (0.7–0.8 ppm), (*E,E)*-3,5-octadien-2-one (2.5–6.2 ppm), acetic acid (137–195 ppm), and hexanoic acid (18–29 ppm) increased with the progress of oxidation; the numbers in the parentheses indicate initial and final concentration of the compounds in the oil in a 6 hour experiment [21]. Hexanol (6.5–20.3 % related to the total level of volatiles), (*E)*-2-butenal (1.3–5.0 %) and acetic acid (3.6–3.8 %) could be identified as the main volatile compounds in the flaxseed oil samples [22].

2.5. Hempseed oil

Hemp is originated from Central Asia and as of today it has spread all around the world. Since the formation of the European Union it is illegal to cultivate hemp crops that exceed a tetrahydrocannabinol (THC) content of 0.2 %. When talking about hemp it is always referred to *Cannabis sativa L.* as seen in Figure 9. Hemp is an annual crop that can reach a height of up to 7m. The fruit of the plant is 3-5mm in diameter and has a nut-like shape, the surface of the nut is green to grey coloured and can have dark spots. (Figure 10) Furthermore, the shell can be quite brittle and contains the greenish seed. The seed itself contains approx. 30-35 % of oil, 20-25 % of protein, but also contains trigonellin, resin and sugar. It is very important to handle the seeds carefully if they are intended to be used for oil production. The seed is pressed preferably cold to conserve all important ingredients and exclude the harm of heat, as in all oil seeds. Usually, the average pressing temperature lies between 40-60°C, with an oil content of approx. 30 % the oil yield is approx. 180-350 l/ha.

The pressed oil itself has a green to yellow colour; the odour is described as aromatic, green-nutty and herbaceous, as well as the flavour is observed as nutty and herbaceous.

Due to the fact that the smoke point is at 165°C the oil is not suitable for frying but is often used in salads or other cold prepared dishes [8].

Figure 9: *Cannabis sativa L.* plant [7]

Figure 10: Hempseeds and the pressed hempseed oil

2.5.1. Special ingredients

2.5.1.1. δ-9-Tetrahydrocannabiol (THC)

THC can be found together with other cannabinoids in the resin of the stems of the female plants, the content depends on the variety of the plant as well as the climate. Cannabinoids are N-free phenolic derivates of the benzopyran, that are derived biogenetically form a monoterpene and a phenol. (Figure 11) The hallucinatory properties are only achieved from the consumption of THC, which cannot be found in hempseeds. Nevertheless, the percentage of THC must be lower than 0.3 % in distributed hempseed oil [8].

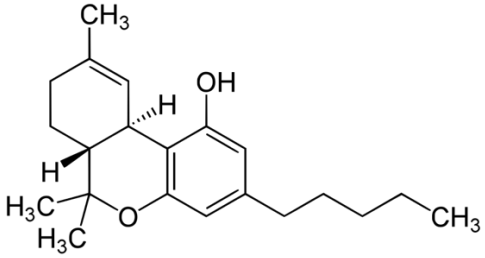


Figure 11: Chemical structure of THC [8]

[7] https://upload.wikimedia.org/wikipedia/commons/f/f9/Cannabis_sativa__K%C3%B6hler%E2%80%93s_Medizinal-Pflanzen-026.jpg

[8] https://upload.wikimedia.org/wikipedia/commons/thumb/4/4c/Tetrahydrocannabinol.svg/2000px-Tetrahydrocannabinol.svg.png

2.5.1.2. ɣ-Linoleic acid (GLA)

ɣ-Linoleic acid is found in hempseed oil in a concentration range of 2-4 % and is an ω-6-fatty acid. (Figure 12) It is very rare to find a natural source of ɣ-linoleic acid; it has only been described contained in human breast milk and in the oil of evening primroses and borage oil. GLA is used in internal as well as external applications for skin diseases such as neurodermatitis. Furthermore, it is used as an anti-inflammatory substance, for instance, in rheumatic diseases. Also, GLA is utilized to decrease the risk for heart attack, cardiovascular-disease, diabetes, and other chronical disease. The human body can produce GLA through the enzyme desaturase from linoleic acid. In the body it works as a biogenetic precursor of prostaglandins with a double bond [8].

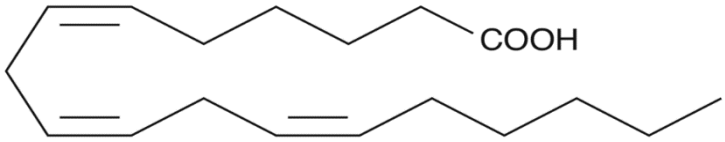


Figure 12: Chemical structure of GLA [9]

2.5.2. Fatty acid composition

Hempseed oil is rich in polyunsaturated fatty acids; an average composition of hempseed fatty acids is given in Table 3.

Table 3: Fatty acid composition of hempseed oil (Source: [23])

	Fatty acid	Weight percentage applied to total fatty acids
C16	Palmitic acid	6.26 ±0.34
C18	Stearic acid	2.72 ±0.03
C18:1	Oleic acid	11.72 ±0.04
C18:2	Linoleic acid	59.96 ±0.23
C18:3	Linolenic acid	19.33 ±0.08

2.5.3. Volatile compounds

Little is known about the volatile compounds of hempseed oil. According to Navas-Hernandez *et al.* [24] terpene compounds can be found in hempseed oil. Also, the authors of this study describe a concentration of 10.1 mg/kg volatile aldehydes, ketones and esters in the investigated hempseed oil samples.

2.6. Legal guidelines and aspects

The Austrian Codex Alimentarius acts as a guideline and does not include any laws or legal restrictions. In chapter B30 of the Austrian Codex Alimentarius, definitions and guidelines for all kinds of edible fats and oils are described. Also, in the same chapter, recommended limits for the peroxide value and the acid number are given. Unrefined oils should not exceed an acid number of 4.0. The peroxide value of unrefined oils should be lower than 10.0. Furthermore, the remaining water content in the edible oil is recommended to be under the value of 0.2 % [25].

[9] https://www.biomol.de/details/CM/gfx/normal/90220.png

Chia seed oil is defined as a novel food and until now there are no exact official quality standards. However, there is a recommendation that the seeds should be cold pressed, the raw material should be of the highest quality possible and as pure as possible. Furthermore, it is advised to control the ω-3-fatty acid content as it is one of the key ingredients in the oil. It is suggested that it should be at least 20 %. Due to its influence on the final product recommended that the harvest conditions are regularly controlled [26]. As mentioned above, the European Commission defines restrictions for some values with regard to chia seed oil. In most cases they state maximum values that should not be exceeded, for example, for α-linoleic acid a minimum value of 60 % is given. The European Commission also imposes a specification for chia seed oil, where oil should be ejected through cold pressing and is made from 99.9% chia seeds from the plant *Salvia hispanica L.* It is not allowed to use any kind of extraction solutions and occurring contamination should be handled using filtration [27].
The cultivation of hemp in Austria and the whole EU is not regulated in the controlled substances law as long as the value of THC in the concerning food does not exceed 0.3 %. The value of 0.3 % was defined, as the usage as a narcotic substance is not possible under this given percentage. The screening of the THC value of the plant is done at the peak of the THC production, which is the end of the blossom. The EU provides a list of seeds that have been investigated considering their THC value and are safe to plant. Only these crops are legal to be used for hemp production. The AGES (Austrian Agency for Food Safety) analysed approx. 130 samples of different foods containing hemp and only one sample exceeded the allowed value for THC [28].

2.7. Maillard and Strecker reaction

2.7.1. Maillard reaction

In the course of the complex Maillard reaction, which is also known as non-enzymatic browning reaction, reducing sugars react with amino compounds. In most types of foods N-glycosides are formed if reducing sugars, proteins, peptides, amino acids or amines are found together, this is eased if the temperature is increased, if minor water activity is found or if the product is stored for a longer period of time. Glucose, fructose, maltose or lactose as well as other reducing sugars are the reactants that are found on the sugar side of this reaction. At the amino acid side of the reaction the primary amino group is of larger importance than the second group, as the concentration is higher of the primary amino group in most types of food [7]. These reactions can lead to different results in the final product. Firstly, brown pigments can be formed that are called melanoidine. Secondly, volatile compounds can be formed, which can be aroma active. On the one hand, this can lead to a desired flavour formation during the cooking, baking and roasting process.
The formed products are having a crucial importance in the aroma and colour development of roasted and baked foods. On the other hand, the development of off-flavour during the storage of a product can be experienced. Furthermore, bitter tasting compounds (such as in the roasting of coffee beans) can be found, these bitter compounds can be desirable or found as off-flavour, formed while roasting fish or meat. In addition, these reactions can lead to the formation of compounds that have strong reducing properties (so called reductones) that can help to stabilize food against oxidative spoilage. However, the Maillard reaction can lead to a loss of essential amino acids such as lysine, arginine or cysteine. [7]. In the first step of the reaction (Figure 13) the sugar compound reacts with an amino group which will produce a glycosylamine compound, through the Amadori-transfer under proton catalysis to an acid stable isomer.

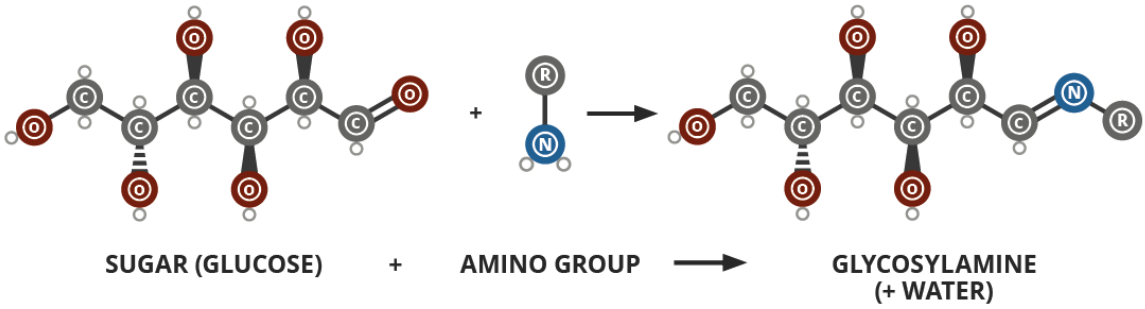


Figure 13: First step of the Maillard reaction [10]

In the second step of the reaction (Figure 14) the formed glycosylamine from step one is rearranged to form a ketosamine. In the final steps this compound can react in a number of different ways to produce other compounds. An H-atom is moved into the 1-position through an eniol-form. Finally, an aldose-derivate is formed and a half-ketal-ring can be formed. The Amadori re-arrangement already leads to brown, high molecular compounds.

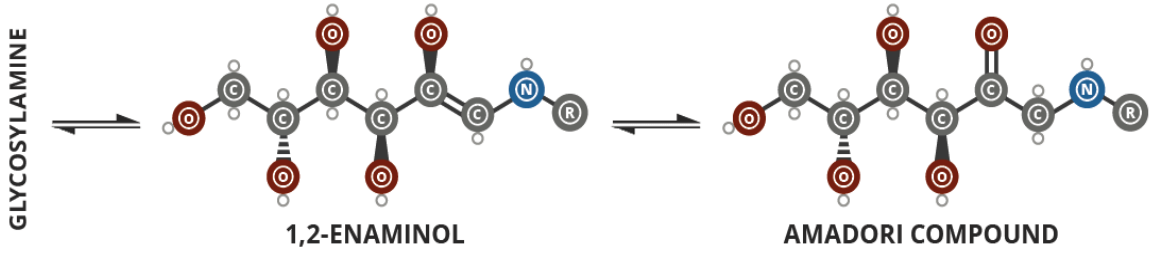


Figure 14: Second step of the Maillard reaction [11]

Different products are formed depending on whether the reaction conditions are alkaline or acidic. (Figure 15) The formed Amadori products are hardly stable, but these products can be transferred into an endiol compound in an alkaline environment and these endiol compounds can be easily put through eliminating reactions. Preferably, the allyl positioned groups are split off, hence a water molecule or an amine residue is eliminated. In the first case, as an intermediate product 3-desoxyhexosone is formed and through the split off of two water molecules hydroxymethylfurfural is formed. In the second case, firstly an 2,3-endiol is formed and an allyl positioned amine-residue split off is preferred. Finally, a 2-desoxyhexoson is formed that can be split into diketons, furanones or furans [5].

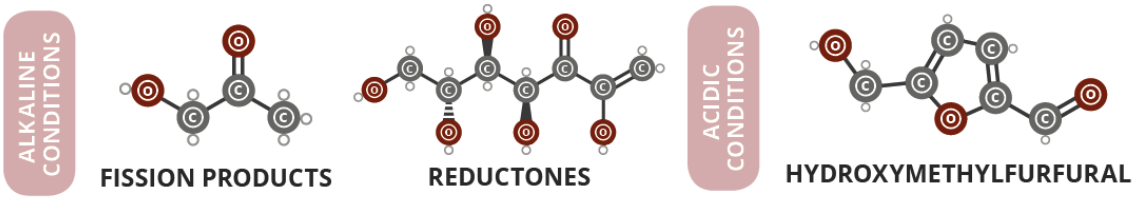


Figure 15: Products that can be formed in the course of the Maillard reaction [12]

In the course of the Maillard reaction, pyrazines can be formed as one of the major products from this kind of reaction. (Figure 16)

[10] www.compundchem.com
[11] www.compundchem.com
[12] www.compundchem.com

As already mentioned, nitrogen atoms gathered from the involved amino acids will react with carbon atoms from the involved sugars. As the formation of pyrazines has been studied in great detail, research shows that the type of the involved amino acids influences the substitution patterns of the formed pyrazines. Furthermore, it was found that glucose yields less pyrazines compared to fructose as a sugar source of the reaction. If the temperature of the reaction is risen to 120-150°C the biggest number of pyrazines can be formed, however already at around 70°C formation of pyrazines could be observed. Another possibility for the formation of pyrazines can be the origin from fermentation. This pattern will be not discussed in detail, as it is not relevant to this thesis [29].

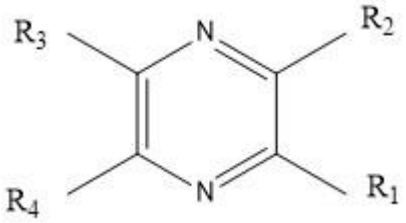


Figure 16: General structure of pyrazines

2.7.2. Strecker degradation

Strecker degradations are reactions between α-dicarboxylic compounds and amino acids. As the α-amino acid is oxidatively decarboxylised to so called Strecker-aldehydes, CO_2 and α-aminoketones are formed. The reactions take place in foods that have an elevated concentration of amino acids and if the reaction conditions are drastic (e.g. high temperature or under pressure). The formed Strecker-aldehydes have a high aroma potential compared to the found amino acids. Important aroma active Strecker-aldehydes are methional, phenyl acetaldehyde, 3- and 2-methylbutanal and methylpropanal, but also other compounds, like H_2S, NH_3, 1-pyrolline and cysteamine, can be formed over the course of the Strecker degradation. These compounds can also be aroma active. Recently it could be shown that oxygen elevates the formation of Strecker-acids [7].

2.8. Lipid oxidation

The process of oxidation of fats and oils is the main reason of spoilage in these products. Oxidation leads to short-chain aroma-active compounds which are mostly responsible for the formation of characteristic aroma-active compounds when found in small concentrations.
If the concentrations increase they can lead to an unpleasant taste and smell. The oxidation process can be divided in the formation of primary and secondary oxidation products.
One of the major challenges for oil producers is to maintain the high quality of the produced oil from the location of the production to the consumer. As edible vegetable oils contain approx. 96% mono-, di- and triacylglycerides that are composed of different fatty acids, those fatty acids (no matter if free or bound to glycerol) are found to be the main reason for oxidative processes happening in the final oil. The reaction of these fatty acids can lead to a broad range of volatile and non-volatile degradation products. The most recognizable changes for the consumer are found in an unpleasant smell and taste of the oil during the oxidation process, but also changes in colour, viscosity, density and solubility can be observed.
As the oxidation continues, essential fatty acids can be lost, as well as vitamins and pro-vitamins are affected by degradation and odour-active compounds are formed.

In the first stages of oxidation hydro peroxides are formed, as those hydro peroxides degrade with the ongoing oxidation, compounds can be formed that can have a toxic potential in higher concentrations. Furthermore, the compounds formed in the course of oxidation can interact with other ingredients of the oil such as amino acids or proteins and can lead to a change in texture and colour. Therefore, these changes may strongly influence the sensory quality, the nutritional value and the consumer acceptance [30].

The overall oxidation process and its velocity is influenced by a variety of factors such as: [7]

- the number of degree of saturation of the fatty acids and fatty acid composition
- the type and concentration of pro- and antioxidants
- partial pressure of oxygen
- surface that is in contact with oxygen
- conditions under which the fats or oils are stored, such as temperature, light and water content
- position of the unsaturated fatty acids in the triacylglycerid molecule

2.8.1. Autoxidation

In most kinds of food, it will take some storage time until the first oxidation products are detectable. It is very typical for autoxidative reactions that the reaction velocity increases over the course of time after the induction period. However, there are also some types of foods which already have a high concentration of prooxidants. In this case, no induction period is experienced. The duration of the induction period and the oxidation velocity depend on the fatty acid composition of a fat or oil. If there are more allyl groups found in the fat or oil the induction period will be shorter and the oxidation will proceed faster. The following ratio 1:100:1200:2500 (which is the ratio of oxidation velocity) on the basis of peroxide formation and the number of allyls for stearic acid: oleic acid: linoleic acid: linolenic acid was found [7]. Stearic acid has no allyl groups, oleic acid has one, linoleic acid has two and linolenic acid has three allyl groups. This leads to the conclusion that high amounts of polyunsaturated fatty acids in edible oils result in fast autoxidation in the storage period. Furthermore, radical intermediate states are needed in this kind of reaction. Only special activated oxygen atoms can be abstracted by the formed radicals [7]. So, in each reaction two main reaction partners are reacting which are unsaturated fatty acids (free or bound to a triacylglycerol molecule) and oxygen. The normal atmospheric oxygen is referred to as triplet oxygen $^3O_2$ which has two unpaired electrons in its ground state.
The resulting reaction is referred to as autoxidation, this free radical chain reaction was first reported by Farmer *et. al.* in 1942 as well as Bolland in 1949 [30]. Up to today it is not clear how these first radicals are formed.

- Initial phase and propagation

$$RH \rightarrow R\cdot + H$$
$$R\cdot + O_2 \rightarrow RO_2\cdot \ (1)$$
$$RO_2\cdot + RH \rightarrow ROOH + R\cdot \ (2)$$
$$RO\cdot + RH \rightarrow ROH + R\cdot \ (3)$$

- Chain branching

$$ROOH \rightarrow RO\cdot + \cdot OH \ (4)$$
$$2\ ROOH \rightarrow RO_2\cdot + RO\cdot + H_2O \ (5)$$

- Termination

$$2R\cdot \rightarrow (6)$$
$$R\cdot + RO_2 \rightarrow (7)$$
$$2RO_2\cdot \rightarrow (8)$$

} stable products

At room temperature, one radical can start the formation of approximately 100 hydro peroxides (start of the monomolecular phase) before a chain termination is reached. Additionally, in the presence of air (oxygen partial pressure above 130kPa) all alkyl radicals are transformed into peroxy radicals through the fast reaction (1). Reaction (2) is the sub step which will determine the velocity of the reaction. If the reaction velocity for the individual steps is measured, it can be seen, that the elongation of the chain growth through abstraction of a water molecular to form a fatty acid molecule is very slow. In reaction (4) the formation of radicals caused by the unimolecular breakdown of hydro peroxides can be seen. This is the cause for the accelerated peroxidation of unsaturated fatty acids as this reaction prolongs auto catalytically. Autoxidation is accelerated by heavy metal ions and haem compounds. Furthermore, this particular reaction step is seen as the basis for the formation of volatile reaction products and the begin of the bimolecular phase. After some time, the concentration of hydro peroxides will reach a certain level and bimolecular reactions of the hydro peroxides will lead to per- and alkoxyradicals (5). Here it should be mentioned that this part of the reaction does not matter in most of the food products, because of the course of fat oxidation the product is already no longer edible before the necessary concentration of hydro peroxides for this particular reaction is reached. The reactions (6) and (7) are only an issue if the food has, for instance, inner parts that are depleted in oxygen. The chain termination is initiated by the collision of two peroxy radicals as seen in reaction (8). It should be further mentioned that the reactions (1) - (8) seen above are only valid for the beginning phase of autoxidation, as secondary oxidation products form, and also partially tertiary oxidation products can be formed, the process gets quite chaotic [7]. Therefore, it is clear that the autoxidation of oils is a reaction that follows an exponential progress, (Figure 17). Every newly formed radical forms again a new radical and a hydro peroxide. Nevertheless, it takes some time until hydro peroxide formation reaches a detectable level. Until today it is not fully understood what the cause for the initial step of this chain reaction is, but it is thought that heat, metal catalysts and visible as well as ultra-visible irradiation are involved. It is well known, that the degree of unsaturation is responsible for the possibility of hydrogen abstraction. This can be explained by the bond strength of the hydrogen of the α-methylene group in the fatty acid molecule. The various fatty acid molecules contain different bond strengths of hydrogen and differences in the rate of lipid oxidation are experienced [30].

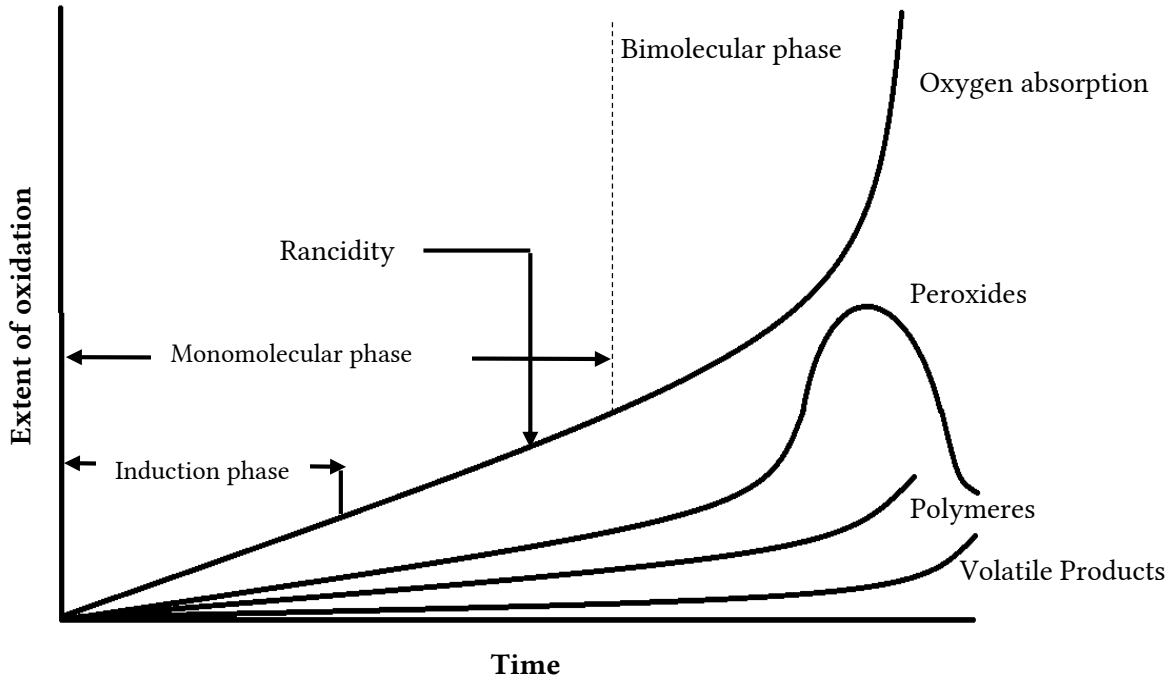


Figure 17: Process of Lipid oxidation [13]

2.8.2. Enzymatic oxidation

This form of oxidation is driven by lipoxygenase which is an enzyme that belongs to the group of oxido-reductases. Those enzymes are found in nearly all living cells and are able to catalyse a reaction between oxygen and a (*Z, Z)*-unsaturated fatty acid from the hydro peroxides, where the main substrates are free fatty acids [30].

2.8.3. Photo oxygenation

The durability of stored oils in presence of light is decreased as an autoxidation of lipids is started. In 1960, Schenk and Koch found that some substances can act as sensitizers. Those sensitizers can be put in two categories: sensitizers from type one will react with the substrate under the formation of radicals which will then lead to autoxidation. In type two reactions the triplet oxygen from the air $^3O_2$ is activated to $^1O_2$. Oxygen in the basic state is found as a triplet molecule $^3O_2$ that prefers one-electron-reactions with radicals. For oxygen in the triplet state it is more difficult to react with compounds in the singulett state, such as fatty acids, to a singlet like hydro peroxides. This is complicated by the spin-barrier of these reactions. But the singlet state is a short-date state compared to the triplet state. Those reactions compete with one another; which reaction will take place is dependent on the structure of the sensitizer and on the structure of the substrate that will be oxygenized. It was found that high oxygen concentration and low substrate concentration will initiate type two reactions [7].

[13] Adapted from [5]

2.8.4. Secondary oxidation products

The formed hydro peroxides cannot be detected by the consumer as they are odour and colourless. The consumers themselves can detect changes in the product, if secondary oxidation products are formed as they come with a great number of odour active compounds and those compounds are detectable from a quite low concentration on. A variety of secondary oxidation products can be formed, mainly odour active carbonyl compounds, aldehydes as well as alkanes and alkenes are formed [7]. The fatty acid composition determines the amount and type of odour active compounds that are formed in the course of oxidation. Especially for oils with high amounts of linolenic acid (such as flaxseed and chia seed oil) the resulting odour active compounds, mostly aldehydes and ketones, have a low odour threshold value. This means that the spoilage of oils with high concentrations of linolenic acid does not only depend on the high accessibility to oxidation, but also on the low odour threshold of the formed degradation products. The found variety of aldehydes is believed to derive from the decomposition of formed hydro peroxides in the course of autoxidation of linoleic acid [7]. Also formed carbonyl compounds with a low odour threshold can contribute to an off flavour. Many pathways for the decomposition of hydro peroxides have been described in literature, but the main known pathway is the haemolytic β-scission of carbon-carbon bonds that leads to oxo-compounds and an alkyl or alkenyl radical, which can be aroma active compounds. Belitz *et. al.* [7], found in 2004 that different fatty acids lead to different degradation compounds in the final product [30].

2.8.5. Repression of the lipid oxidation

There are possibilities to slow down the lipid oxidation or even to prevent those reactions. One possibility is the exclusion of oxygen in form of a vacuum. An addition of glucose oxidase can be used as well to supress the lipid oxidation. Another possibility is the storage at very low temperatures and in darkness. The velocity of the autoxidation can be lowered with these techniques. Also, the addition of antioxidants is possible [7].

2.9. Sensory evaluation

To be able to recognize a sensory impression detected by any of the human senses it is necessary that stimuli are transported via electrical nerve impulses directly to the brain. The processing already begins in the sensory organs and is not confined to the brain. However, in the brain those stimuli are further processed in specific regions and are coupled with different experiences. Based on those experiences stimuli are interpreted and the stimuli is perceived as picture, movement, sound, taste, temperature or odour. Stimuli and their interpretation and perception is always coupled with psychological parameters such as experience, skill and emotions. The combination of these factors makes it possible to distinguish between five basic tastes or over 10.000 odour active substances. In sensory evaluation the sensory quality of e.g. foods is measured, analysed, evoked and interpreted using the human senses. Sensory evaluation can provide quantitative and qualitative data and insights into the specific sensory characteristics of a certain food. However, the impression of the human senses can be subjective sometimes and influenced by various external factors. To eliminate this factor highly trained sensory expert panels are used.

2.9.1. Sense of smell

The sense of smell or olfactory sense is a chemical sense because olfactory perceptions are evoked by soluble and volatile chemical compounds. Those volatile chemical compounds can provide a great variety of information about the food we are about to taste.

Yet, olfactory perception is heavily influenced by the education and experience of every human. The olfactory sensory cells that are responsible for the reception of the sensing are found in the olfactory mucosa. Receptor molecules in the membranes of the sensing cells can recognize a chemical sensing and help to transport these sensing impressions as an electrical signal through nerve fibres into the olfactory bulb. A principle of the perception of smell can be seen in Figure 18. Odour can be perceived in two different ways: One possibility to perceive an odour impression is over the receptors and sensory cells in the olfactory mucosa in the nose, which is called orthonasal smelling. The second possibility to perceive an odour impression is over the oral cavity and the olfactory epithelium found in the oral cavity, which is called retronasal smelling. As the nasal and oral cavity are linked through an opening, odour impressions after swallowing food and breathing out are more intense as the compounds can travel over the pharynx further on to the olfactory mucosa. In general, it is quite hard for humans to describe the odour perceptions and to put those perceptions into words. As both of the nasal wings are not working constantly, it is a sensory practice to perform sniffing to get a more intense odour impression of the product as more air can be transported through the nasal wings. If sniffing is performed small eddies are formed in the upper nasal area and the odour impression is intensified [31].

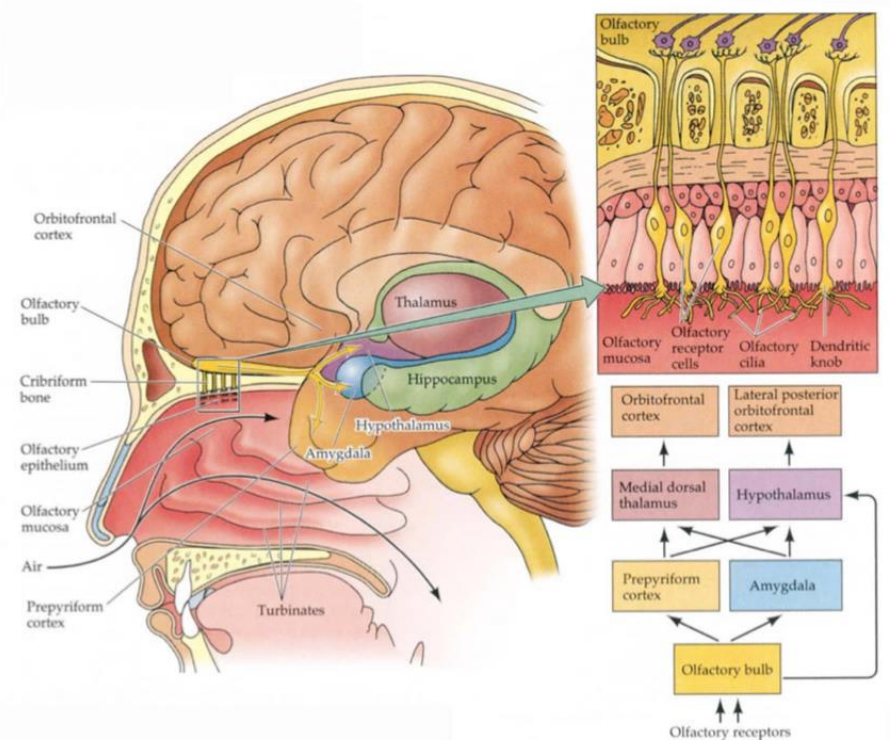


Figure 18: Olfactory system of the human body [14]

2.9.2. Sense of taste

The sense of taste is a chemical sense because the water- or saliva-solvent chemical compounds in the food are only registered if they are directly touching different regions on the tongue. Only through chewing and the added saliva all odour active compounds of different kinds of foods can be set free. For humans it is possible to distinguish five basic tastes which are sweet, salty, sour, bitter and umami. Umami is described as a savoury-spicy taste impression and can be found in food that is high in protein and is evoked from the amino acid glutamate. To simulate the sense of taste the substance has to be soluble in saliva or involved in the salivation.

[14] http://www.allpsych.uni-giessen.de/thomas/teaching/pdf/Allg2008/02-sinnesphysiologie.pdf

The compounds that are dissolved in the saliva interact with the taste buds and trigger a stimulus at the sensory cells of taste. Taste buds are found in different kinds of papillae that which are spread throughout the edge of the tongue, the tongue base, on the palate, the throat and the epiglottis. Looking closer at the papillae different kinds of papillae can be distinguished. On the tip of the tongue small fungiform papillae can be found, on the ground of the tongue bigger vallate papillae and leaf shaped papillae can be found. About half of all taste buds are found in the vallate papillae. A principle of the gustatory system can be seen from Figure 19. The amount of taste buds on the tongue can differ, a super taster can have up to 1000 taste buds per cm^2 on the tongue. Whereas a normal consumer has around 200 taste buds per cm^2 on the tongue, some people can only have around 12 taste buds per cm^2 on the tongue and will need quite heavy stimuli to taste or smell something. The number of taste buds decreases with increasing age, but some loss can be compensated with the sensory memory. New research shows that it is possible to taste the five-basic taste impression over wide spread areas of the tongue, but especially on the edges of the tongue. In sensory testing's often small portions of food are put in the mouth and after judging these portions are spit out again. It is very important to neutralize the taste buds and get rid of remaining food residues in between tastings with water or white bread [31].

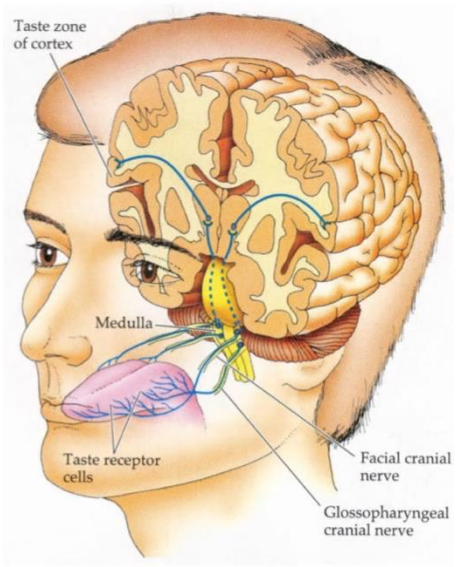


Figure 19: Gustatory system of the human body [15]

[15] http://www.allpsych.uni-giessen.de/thomas/teaching/pdf/Allg2008/02-sinnesphysiologie.pdf

3. Applied Methods

In the following chapter the main equipment and their principles and applications are described. Furthermore, the used methods and devices are given including the sample preparation. As this thesis discussed two main topics the following chapters will be split. Part one will focus on the evaluation of a rapid test method for the quick and reliable determination of fat classification numbers. The second part will deal with the investigations concerning the roasting and pressing process of the different oil seeds. This is followed by the storage of the freshly pressed oils until the final filling, finished by the examination of the storage period of the three different oils within and over their best before dates.

3.1. Evaluation of a rapid test for the determination of fat classification numbers

At oil mill Fandler a fast determination method for fat classification numbers is used. To prove the applicability of the rapid test on cold pressed oils, evaluation of the rapid test has to be performed by comparing the obtained results with those obtained from standardized methods.
cdR Food Lab Junior (CDR, Florence, Italy) [32] is a photometric analyser which uses LED emitters, reading cells and 37°C thermostatic incubation cells to perform different readings. The analyser is already pre-calibrated. Furthermore, no calibration or maintenance is required. It is possible to analyse different fat classification numbers, these numbers are determined using low toxicity, single use and pre-vialed reagents. They have a shelf life of 1 year and some of them, like the acid number kit, should be stored at cool temperatures. Pipettes are used for sample collection. According to the product description, it is possible to analyse all different kinds of fats and oils using the cdR Food Lab Junior. To determine the acid number, the principle of decreasing colour if a free fatty acid of the sample at the pH of < 7.0 reacting with a chromogenous compound is used. The decrease is then read at 630 nm and is proportional to the free fatty acid concentration of the sample, which is given as % of oleic acid [33]. For the determination of the peroxide value the principle of R-O-O-R peroxide value oxidizing Fe 2^+ ions and the forming of Fe 3^+ ions resulting from oxidation is used as they form a red complex when grouping. The colorimetric adsorption is measured at 505 nm and is directly proportional to the concentration of peroxide value in the measured sample, the results are given in meqO_2/kg [34]. To determine the anisidine value the aldehydes that are formed via secondary oxidation of the fat matrix and further reaction with the p-anisidine and the resulting change in the absorbance is measured at 366 nm [35].

3.2. Fat classification numbers

3.2.1. Acid number

The acid number is a measure for the content of free acids in fats and oils, besides the fatty acids also mineral acids are determined. It is used to review the purity of the tested fats and oils and sometimes it is possible to conclude pre-treatments or decomposition reactions. Raw and unrefined oils usually show an acid number lower than 10, refined oils show an acid number lower than 0.2. The acid number is defined as the amount of potassium hydroxide in mg, which is needed to neutralize the free acids in 1 g fat (or oil). The principle of the method is the dissolution of the sample in an organic solvent and the present acids are titrated against phenolphthalein with potassium hydroxide [36].

3.2.2. Peroxide value

The peroxide value is a measure for peroxided bound oxygen in fats. As a primary oxidation product, especially hydro peroxides are formed besides other peroxides due to oxidation processes (autoxidation). Therefore, the peroxide value gives an idea of the degree of oxidation of the sample and can help to determine the spoilage of the fat. In this context, it is important to consider that with proceeding oxidation the peroxides degrade and the peroxide value will decrease again. The peroxide value is defined as the number of active oxygen which is found in 1 kg of sample and is stated in 1/8 mmol/kg. If the peroxide value is multiplied by the equivalent mass of oxygen (=8) the mg of active oxygen per kg of sample can be calculated. The sample is dissolved in a mixture of chloroform and glacial acetic acid and is mixed with a potassium iodide solution. Because of the reaction with the peroxide groups, iodide is set free. The amount of iodide is specified with titration using sodium thiosulfate solution [36].

3.2.3. Anisidine value

The anisidine value is describing the amount of α, β-unsaturated aldehydes that are found in a fat or oil sample. Often α, β-unsaturated aldehydes that are formed in the course of autoxidation are represented by 2-alkane and 2,4-dienale, but also oxo-compounds bound in the triglyceride bond are covered. The anisidine value gives an idea of the history of the oil or fat sample and can also sometimes give an idea for the durability of the sample [36].

3.2.4. Totox value

The Totox value is used to give information on the oxidative degradation of the investigated oil. It can be helpful to determine this number to give additional information on the degree of oxidation of oil. The value is a calculated value using the experimentally determined peroxide and anisidine value for the sample. The Totox value takes the primary and secondary breakdown products of the oil or fat sample into account. As the value captures the analytically determined reaction products that can be formed in different states of the autoxidation. Yet as the experimental determination of the needed values is quite complex it should be mentioned that the Totox value has only an orientating character [36].

3.3. Investigation of the roasting, pressing and storage process of selected oils

3.3.1. Sample preparation

If a sample should be analysed using gas chromatography sample preparation has to be performed. This includes the solution and/or extraction of the analytes, detaching of interfering substances and purification and enrichment of the analyte. Before the measurements can be performed, the concentration of the sample has to be either increased or decreased in order to have the suitable concentration for the selected determination range. Sometimes the analytes have to be transferred into a suitable solvent, e.g. if a HPLC analyses is used. For GC analysis solvents with a low boiling point are preferred, as they evaporate fast in the injector and give a small solvent peak in nonpolar standard phases. Often an internal standard is added to the samples before the sample preparation that passes through the whole preparation process. Ideally the chosen internal standard has similar physical and chemical properties to the analyte [37]. To be able to investigate the volatile compounds of a food sample, adequate sample preparation has to be carried out prior to the gas chromatographic analysis. Therefore, the odour active compounds have to be set free into the gaseous phase if a headspace technique is used.

Considering the odour active compound analysis using headspace solid-phase micro extraction it is very important not to alter the odour active compounds prior to the analysis [38]. Furthermore, it is crucial to choose a weight of sample where the volatile flavour compounds can be detected, as they are found in low concentrations between µg/kg and ng/kg. The volatile compounds should be extracted as mildly as possible to prevent the formation of products through reactions. Especially in products where the aroma is formed through the Maillard reaction the isolation of the flavour compounds should be done at temperatures below 50°C. If the temperature would exceed 50°C additional odour active compounds such as thiols, through the reduction of disulfides, could be formed. Some fats and oils can contain volatile and non-volatile hydro peroxides that can fragment at temperatures over 40°C [7]. The analysis of the components of the oils was carried out with headspace solid-phase micro extraction (HS-SPME) gas chromatography - mass spectrometry (GC-MS). Headspace solid phase micro extraction is often used in flavour chemistry as it is a fast and solvent less alternative to other sample extraction methods. The analytes in the sample establish a balance between the sample matrix, the headspace over the sample and the used polymer-coated fused fibre. Using headspace-GC the gas phase over the sample is transported into the gas chromatography unit at room temperature (or a set temperature). The volatile analytes are separated from the semi volatile substances that could interfere with the analyses. Using this technique, the injection can be combined with the sample preparation. The used fibre is a glasfibre built into a modified microliter syringe and acts as an absorber or adsorber depending on the used fibre coating. This glasfibre are modified, e.g. with coatings similar to those used on stationary GC phase such as polydimethylsiloxane (PDMS). The analytes are concentrated on the fibre and delivered rapidly to the chromatography column. These analytes are desorbed from the used fibre in a following chromatography column, as seen in Figure 20 [37].

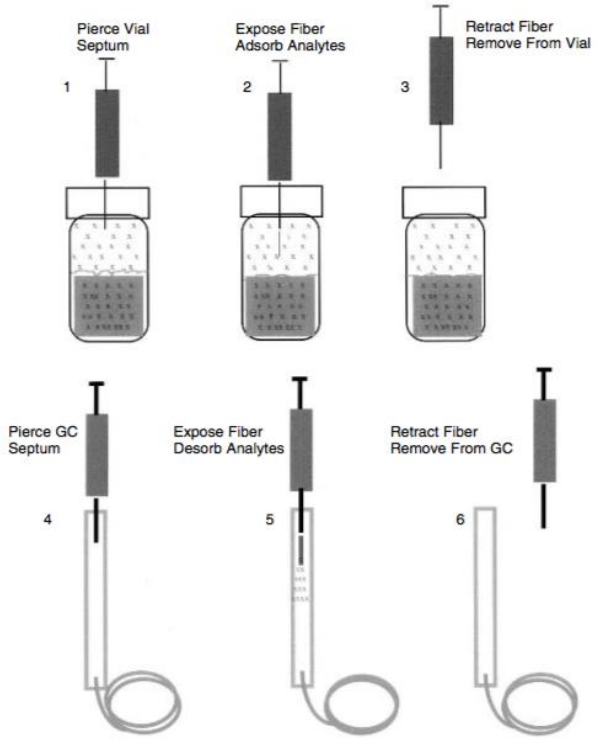


Figure 20: Steps in a SPME headspace analysis: 1–3, extraction; 4–6, desorption [16]

[16] Supelco, Inc.

3.3.2. Gas chromatography

The principle of gas chromatography (GC) is the separation of a compound between a mobile and a stationary phase. Those two phases are not miscible, the separation is based on the different distribution characteristics of certain compounds. Over the course of a chromatographic run the partition equilibrium is constantly maintained. If gas chromatography is used, only compounds that can be vaporized without being harmed can be analysed. Gas chromatography is therefore used to determine small, nonpolar and volatile compounds. Complex samples can be analysed and often detectors with high sensitivity are used, making detections in small concentrations, e.g. in in trace analytics, possible. As mentioned above a mobile and a stationary phase are used in a GC. The mobile phase is used to transport the compounds through the column and is crucial for the separation efficiency. Mostly helium or hydrogen are used. As a stationary phase nonpolar or polar column are used. If nonpolar columns are used the compounds are mainly separated according to their boiling point. However, if a polar column is used the compounds are separated according to their boiling point and also their polarity [37]. Often a GC is coupled with a mass spectrometer to identity the analysed compounds which is then referred to as GC-MS. To make sure a separation is possible the single components must be dissolved, absorbed or adsorbed by the stationary phase. Depending on the chemical properties of the compound and the phase, the phase can act as a solvent agent or as an adsorbing agent.
This means that the single compounds are more or less likely to be held back by the stationary phase and will then reach the detector at the end of the analytical column at a shorter or longer period of time carrier gas flow [36]. A principle set up of a GC-MS can be seen in Figure 21. Another important component of the set-up is the temperature of the column which can be used to optimize the separation of the single compounds. This is also the reason why the column is placed in an oven. If a GC with a nonpolar stationary phase is used the compounds are separated according to their boiling points. A rise in temperature can lead to shorter retention times for compounds with a lower boiling point, which means that a shorter overall analysing time is needed. However, a rise in temperature can also lead to a lower resolution. As the optimal temperature for all desired compounds is mostly not in the same temperature range, so called gradient elution is applied. The temperature of the column is increased constantly over time. Nowadays, these temperature programs can be put into the software of the gas chromatography that will control the GC unit and the column oven [37].

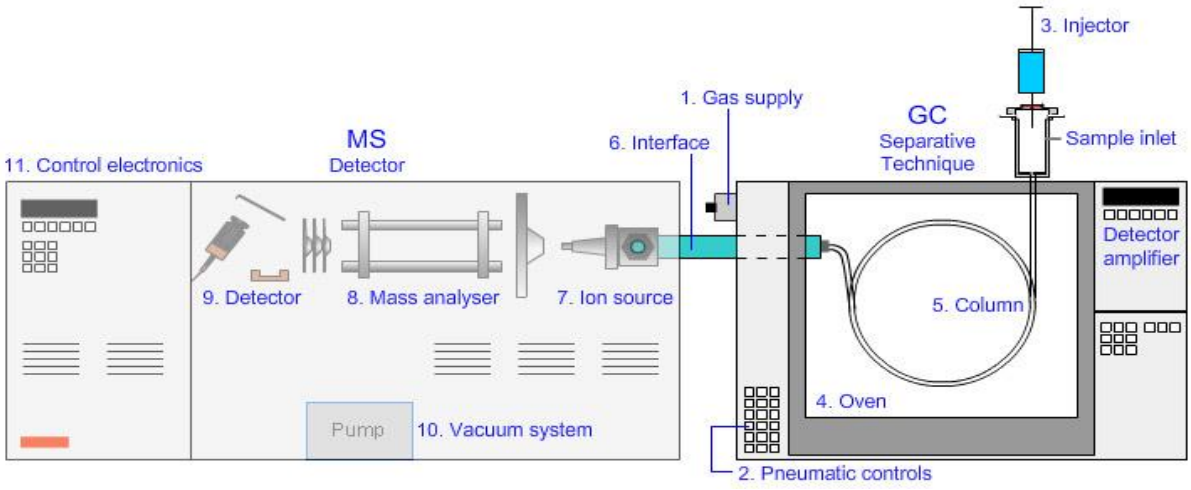


Figure 21: Principle set up of a GC-MS

As mentioned above, as a stationary phase a solid can be used and the gas chromatography would be termed as Gas-Solid-Chromatography (GSC). Also, liquids can be used as a stationary phase, which is the standard technique at the moment, termed as Gas-Liquid-Chromatography (GLC). The liquid can be applied to the inert carrier as a thin film or the column can have a thin film of liquid. Either way temperatures above 400°C are possible depending on the used column with this technique and a lot of different kinds of liquid phases are available, giving a broad spectrum of applications. It can be used to investigate gaseous or fully vapourable compounds. As with the GC typically volatile substances are analysed, these are mostly small and nonpolar molecules. Therefore, the polarity of the stationary phase is chosen according to the polarity of the analytes. Widespread are nonpolar stationary phases that consist of poly(dimethylsiloxan)- and poly(5%-diphenyl-96%-dimethylsiloxan)-phases. The analytes are separated according to their boiling point. The substances with a lower boiling point will elute earlier and the substances with a higher boiling point will elute later.
Often stationary phases with polar characteristics can be used, to make the polarity an additionally separation criteria. This technique is often used to separate compounds that have a quite similar boiling point. Very polar stationary phases are then used to separate polar molecules [37]. Nowadays, capillary columns are often used, the mostly used columns are glass-silica-capillars with an inner diameter of 0,2-0,3 mm, a length of approx. 10-50 m. Capillary columns can also be further distinguished into different types. (Figure 22) Four main types are discussed here:

- WCOT-Capillars (Wall Coated Open Tubular): These capillars are thin-film capillars which have the separating liquid brought onto the pre-treated, relatively smooth inner surface. This capillary type is most often used [37].
- PLOT-Capillars (Porous Layer Open Tubular): Here absorbent particles are fixed to the inside of a fused silica tubing, which will hold the absorbents in place while using. These columns are mostly used for the separation of small molecules such as permanent gas or light hydrocarbons [39].
- SCOT-Capillars (Supported Coated Open Tubular): These capillars are thin-layer capillars which have a substrate coated surface (for instance with silica gel). This leads to an enlarged and rough surface of the capillars, which is then covered with the liquid phase [37].
- Chemical bond phase (CB, Chemical Bond): The separating phase is bond chemically (covalent) onto the glass surface. Bleeding of the column is nearly impossible with this type and the capillars can be rinsed with solvent if needed [37].
- Fused Silica Capillars: Capillars are furnished with thermostable plastic sheeting and become nearly shatterproof. These type of capillars have mostly replaced the not coated glass capillars [37].

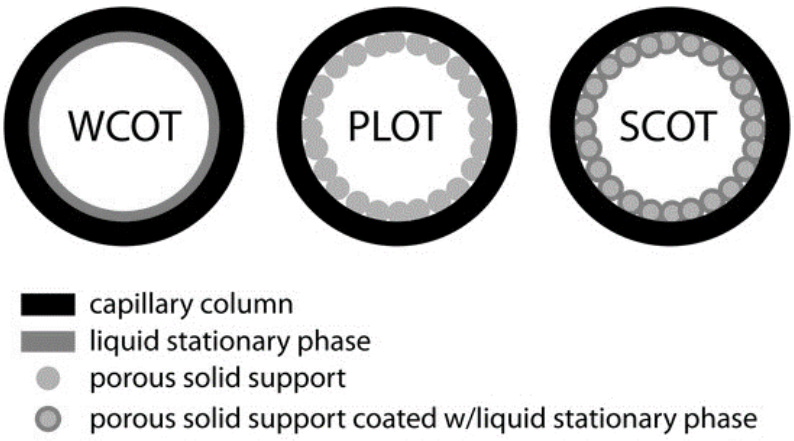


Figure 22: Different types of mostly used GC Columns [17]

Nowadays, capillary columns are used mostly rather than packed columns. As the sample has to reach the column an injection port as a sample placement is needed. Mostly the volatile compounds dissolved in a liquid extract or liquids are transferred using an injection syringe through a self-sealing rubber membrane to the injector and the compounds are vaporized. The self-sealing rubber membrane is often referred to as septum. The amount, often between 0,5 µL to 2 µL, that is brought to the column can be directly seen at the scale of the syringe.
The amount of sample that is used is mostly depended on the type of column that is used, on the amount of stationary phase in the column, respectively the film thickness of the thin-film-capillars, as well as on the solubility and polarity of the compound and the solvent in the stationary phase and the temperature and on the sensitivity of the detector. If capillary columns are used mostly a split injection is done especially if liquid techniques are used. If trace analysis is done split less injection is used mostly. Another important aspect of the GC analysis is the temperature of the column and the resulting separation of the compounds. Here it is crucial to program the increasing temperature as a temperature program, because this can give the optimal boiling range for every compound in the sample and will give ideally a separate peak for every compound. On the one side, at a constant temperature compounds with a low boiling point will eluate quite shortly after one and another and peaks can overlap. On the other side, compounds with a high boiling point often give a quite shallow peak or sometimes are kept back by the column completely. Furthermore, the film thickness of the column is another important factor in the separation of the compounds using a GC-MS. Thicker films are usually used for volatile compounds and thinner ones for high-boiling compounds. With films with a coating thicker than 1 µm extremely low boiling compounds, such as volatile halogenated hydrocarbons can be separated. As the capacity increases greatly with a thicker film it is usual to use start temperatures above room temperature, the starting temperature always depends on the column itself. However, if a thick column coating with more than 1 µm is used sever column bleeding can be a problem at elevated temperatures. For other compounds that should be analysed coatings with a thickness of approx. 0.1 µm are generally used in GC-MS. These kind of thin film columns will give rapid peaks and no column bleeding will be experienced at higher temperatures. Also, if thinner films are used the elution temperature of compounds decreases and compounds with higher molecular weights can be analysed. To summarize, the benefit of an increased film thickness of the column is the improved resolution of volatile compounds, but the disadvantages are the increased analysis time as well as the experienced increase in the elution temperature [40].

[17] http://delloyd.50megs.com/images/capillary1.jpg

Detectors are used to distinguish compounds based on the determined changes in the physical properties of a compound. Different types can be used here as well:

- Flame Ionisation Detector (FID)

As this type of detection was used determining the fatty acid composition of the oils it will be described in detail below.

- Thermal Conductivity Detector (TCD)

The measuring principle is based on the measurement of the difference of the thermal heat conductivity between a sample gas flow and a reference gas flow. As the samples are not destroyed when they are detected the TCD can be coupled with other detectors, then a so called tandem detection is applied. As TCD detection, compared to others, shows a low detection limit it is not suitable for trace analytics. It is often used for the detection of permanent gas, nitrogen, carbon dioxide or inert gas.

- Electron Capture Detector (ECD)

Nitrogen flows as a carrier gas through the detector and is ionised using β-radiation from a radioactive source. So-called "slow electrons" are produced with this method, which are flowing towards the anode and form the base flow. If the carrier gas flows through the detector with substances that have a high electron affinity the electron flow is lowered because of the absorption of the carried electrons. The amount of which the flow is lowered is the measured value that is proportional to the found compound. This kind of detector is often used in trace analysis as they have a high response for a certain substance group. However, the detectors have a quite small linear range [36].

3.3.3. GC-MS (Gas Chromatography with Mass Spectrometry)

Often in food analysis gas chromatography is connected to a single-quadrupole-mass spectrometer. Using a mass selective detector, the molecules are transferred into ions, that will then be separated according to their mass-charge-ratio using an electric or magnetic field. A quadrupole-mass spectrometer (Figure 23) has a cathode which is heated directly and emits electrons that will be accelerated in an electric filed and shot into the ionization room. A small amount of the molecules in the gaseous phase in the ionization room will be ionized through an electron impact. The type and amount of the occurring ions depends on the energy of the bouncing electrons; the ionization process requires a minimum energy which is referred to as "appearance-potential". The ionization process increases from around 50 to 150 eV depending on the type of gas that is used. Most reference data are determined at 70 eV. At higher electron energies besides simply charged ions also multiple charged ions can be found, though with lower yields. At the ionization of molecules with increasing complexity a great variety of fragment ions occur, the type and amount of these fragment ions can be characteristic for certain molecule types. A spatial inhomogeneous and temporal variable electrical field is used to separate the ions according to their different mass/charge ratio [41].

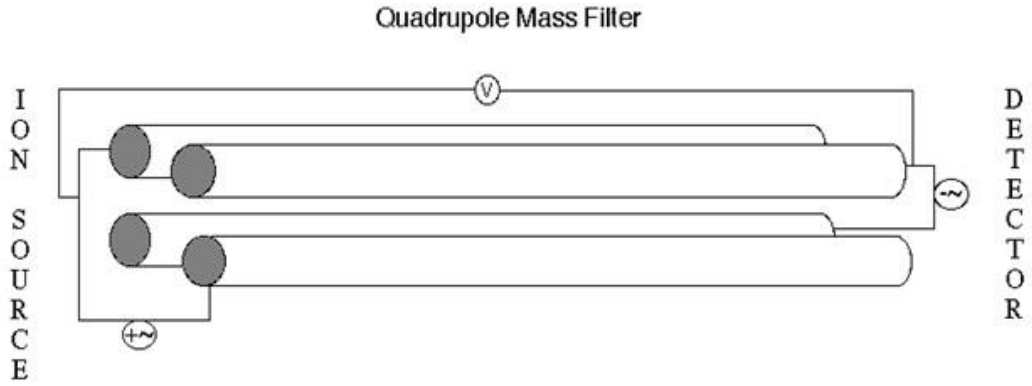


Figure 23: Principle of a quadrupole mass spectrometer [18]

3.3.4. GC-FID (Gas Chromatography- Flame Ionisation Detector)

The fatty acid composition was determined using an GC-FID (Gas Chromatography- Flame Ionisation Detector) technique. The FID detector (Figure 24) is often used for analysing food samples. Molecules are ionised in a hydrogen flame and the resulting ion current is measured, amplified and registered. Usually hydrogen and compressed air are needed as burnable gases. Furthermore, the detection is depending on mass current which means that the signal is higher as more substance per time unit is ionised by the flame [36]. The FID detector is the most often used detector in combination with gas chromatography and is a very general applicable detector. Using a FID, the flow from the column is lead into a small air-hydrogen flame. In the hydrogen-rich part of the flame (reducing conditions) nearly all carbon compounds are transferred into methane (CH_4). The resulting methane is burnt through radical reactions in the oxygen-rich part of the flame (oxidizing conditions). The resulting CH^+ radicals can react with stimulated oxygen compounds and form CHO^+ ions. Above the flame the collector is found, and electrical potential is created between the flame and the collector. In a manner, that the collector is negatively charged against the flame burner. The formed CHO^+ ions are therefore attracted by the collector and an ion flow is created, which is proportional to the ion number and is registered as the detector signal [37]. The typical detection limit of a FID is at around 1pg /s [42].

[18] Adpated from https://i.pinimg.com/originals/ce/4e/6a/ce4e6a47d8c18db86103cb4ea4dd6c32.gif

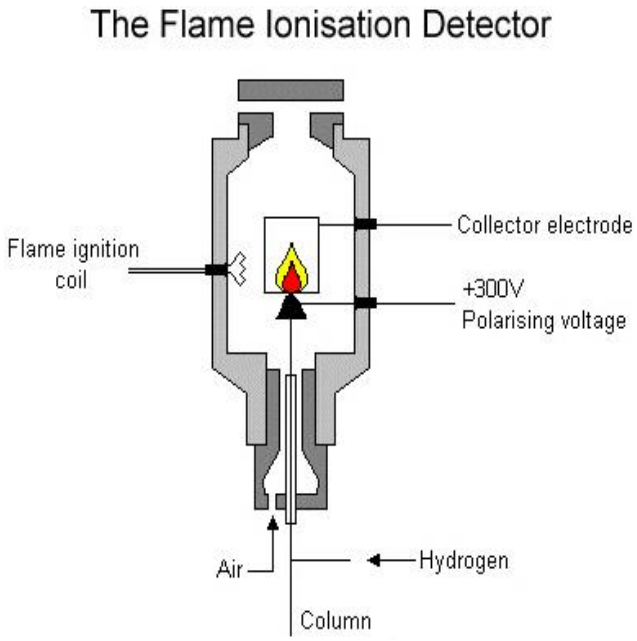


Figure 24: Principle of a flame ionisation detector [19]

3.4. Identification and validation of the detected compounds

To identify a certain compound at first the given mass spectra of the compound has to be corresponding to the same compound in a validated database. Another possibility is to measure adequate reference substances for comparison, also it is possible to evaluate the determined mass spectra based on the knowledge of the researcher, which is referred to as interpretation of mass spectra. Secondly the retention indices that have been calculated as given below should be corresponding to the retention indices of this compound in a certain database, or retention indices can be compared to databases that have been obtained by the research facility. To gain the retention indices reference compounds have to be measured with every performed sample measurement. Furthermore, a comparison to primary literature is possible.

3.4.1. Calculation of Retention Indices (RI)

The retention indices (RI), also known as Kováts-Indices, are used to compare the elution behaviour of volatile compounds on different types of gas chromatography at a given polarity of the stationary phase. The RI makes the given retention times independent form the used device. Ervin Kováts developed this system in 1958. Lateron van den Dool and Kratz modified the original formula and nowadays, the linear temperature programmed RI is calculated with Equation 2 [43].

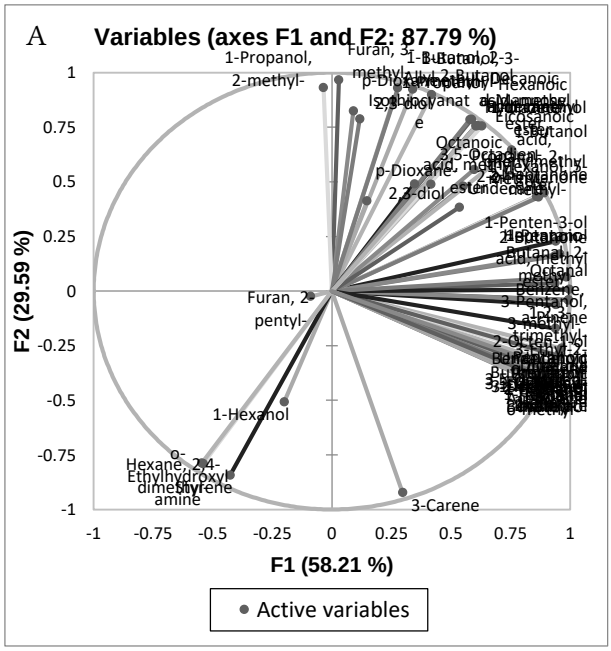
$$RI = \frac{100\,(T_x - T_z)}{(T_{z+1} - T_z)} + 100z \qquad \textbf{Equation 2}$$

T_x	retention time of component x
T_z, T_{z+1}	retention time of n-alkanes, bracketing of component x
z, z+1	number of carbon atoms in n-alkanes z or z+1

[19] http://teaching.shu.ac.uk/hwb/chemistry/tutorials/chrom/fid.gif

3.4.2. Statistic evaluation

For the evaluation of the obtained results, especially from the roasting and pressing process of the investigated oils the data was processed using XLSTAT. XLSTAT is a statistic program that is used as a plug-into Microsoft Excel. In this case Microsoft Excel 2016 was used with XLSTAT. The program was used to perform a principal component analysis (PCA). PCA is a statistic tool to investigate numerical data to provide a quick graphic view of the correlation of a dataset and the correlations between the variables. Furthermore, it is possible to visually see and analyse observations and also gather a set of uncorrelated factors in a given dataset. It needs to be stated that the PCA is an exploratory statistical tool and results have to be interpreted with caution to avoid any misinterpretations. In brief, some guidelines to interpretation of the gathered graphics will be given. The first graphic that is given is a so-called correlation circle where the initial variables are projected in the space of the factors. Hence, if two variables are far away from the centre but quite close to each other they are significantly positively correlated. If they are orthogonal they are not correlated and if they are on opposite sides they are significantly negatively correlated. As an example, the correlation circle of the dataset obtained from the flaxseed oil samples is given in Figure 25.



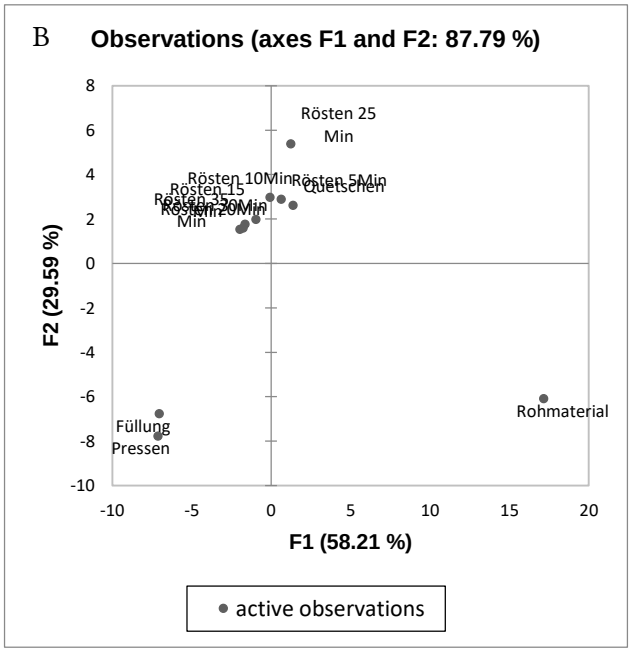


Figure 25:Correlation circle obtained with the XLSTAT Software for the evaluated flaxseed oil samples (A)

Figure 26: PCA as a 2D Map obtained with the XLSTAT Software for the evaluated flaxseed oil samples (B)

Additionally, Figure 26 shows the Principle Component Analysis as a 2D map with chosen variables in which one is able to identify trends. Here the same rules for interpretation as to the correlation circle apply. It is also possible to get biplots from XLSTAT which simultaneously display the observations and variables of the PCA. In this case the analysis was carried out using the PCA tool of the XLSTAT software giving a linear Pearson correlation of the selected data and generating a biplot [44]. To evaluate the performed sensory evaluation using QDA the obtained data was processed using a spider web profile as well as a PCA. A spider web profile or also spider web plot can be used to demonstrate values graphically that are put in the same predefined categories, such as in this study the different odour impressions for hempseeds and hempseed oil.

For every category an axis is defined, all axes are in the same orientation and the axes are put in a circular formation. The higher values lie outside of the centre in the spider web plots and the lower values are positioned more towards the centre. The obtained values relate to lines to give a better visibility. For every category a colour is defined, sometimes the obtained areas are also filled. To gain a spider web plot a least three categories must be defined. Also, there should be not more than 10 axes, but at least 4 axes [45].

3.4.3. Evaluation of the obtained GCMS data

The data of the GCMS investigation of the samples of the roasting, pressing and storage process of all oils were processed using the Enhanced Data Analysis Software provided by Agilent. Additionally, the Signal-to-Noise-Ratio (S/N ratio) was determined for the roasting and pressing process of chia seeds, flaxseeds and hempseeds. Therefor the S/N ratio was determined manually within this software. Calculating the ratio of the peak of interest to the noise in the background. The limit of detection (LOD) was set at < 3, the limit of quantification was set at < 10. If a S/N ratio below or at 3 was detected, the compound was labeld as not detectable (n.d.) and was not further quoted in any tables. If a S/N ratio between 3 and 10 or exactly at 10 was detected, the compound was labled as not quantifiable (n.q.) and was labled n.q. in further tables. For the compounds determined as not quantifiable, a quantification limit was determined individually for either the roasting and pressing process for all three oilseeds and oils. This quantification limit was used for further calculations and tables. As basis of this determinations the "Strategy for determination of LOD and LOQ values –Some basic aspects, Talanta 119 (2014), 178-180" from Jozef Uhrovcik was used. The basis of the LOD and LOQ is visualized in Figure 27.

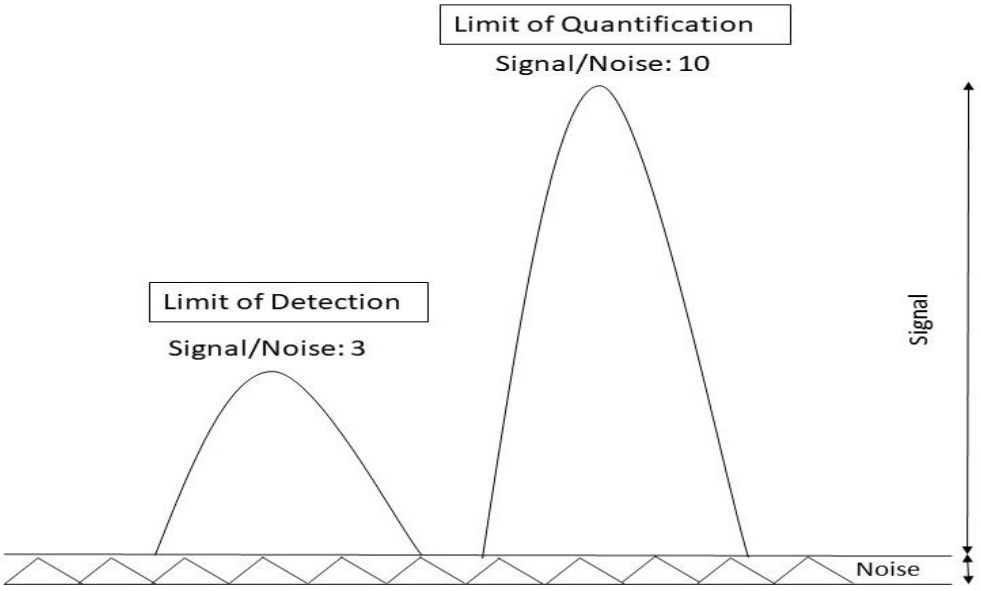


Figure 27: Limit of detection) and Limit of quantification and reference peak[20]

[20]: Adapted from http://slideplayer.com/slide/6184349/

3.5. **Sensory evaluation techniques**

One possibility to do sensory evaluation is with a sensory panel that is especially trained for sensory evaluations. For analytical evaluations difference tests as well as descriptive tests can be done. The descriptive tests are done to evaluate the human perception of a certain product, but they can also be used to observe a storage process or the qualitative standards of a certain product. Often this kind of tests are performed at first with a specially trained sensory panel that has also a trained vocabulary in sensory science.

- **Classic descriptive tests** are mostly done as a profile test that gives a description of sensory product characteristics, is based on the sensory perception of a trained and qualified individual, as defined by Stone and Sidel in 2004. Testers for this method must be trained beforehand in the specific characteristics of the product and the special vocabulary that is used for the qualitative description. Often also reference samples are included to standardize the perception of the testers.
- **Simple descriptive evaluation** (DIN 10964-2014) has the aim to describe some or all product characteristics (such as appearance, odour, taste, texture). It is possible to do this testing with trained or untrained individuals; the only criteria are that the testing individual can describe the sensory perceptions in an applicable and understandable way. It is either possible to give a defined list of appropriate descriptors or it can be freely selectable. However, the chosen vocabulary must be free of hedonic rating, like nice or awful. In the same basic manner, but with more intensive and specific training a **conventional profile** (DIN 10967-1-1999) can be used to describe products in their qualitative and quantitative characteristics. Using this technique, a list of terms is defined beforehand, and the product is rated with these terms and their intensity. Individuals that take part in these testing's must be trained very specifically and precisely and to give a valid outcome at least six individuals should take part in the evaluation [47]
- QDA stands for **Quantitative Descriptive Analysis** and was described by Stone as a method that should give the possibility that products can be evaluated by trained individuals. Additionally, some general features have been defined. In a QDA analysis all sensory impressions should be captured by the analysis. Also, multiple products should be tested and not one isolated product. The QDA should be done with at least 10 testers but not more than 12 testers. The individuals for the analysis should be selected and trained beforehand. Furthermore, in a QDA the testers rate the different properties of the product on an unnumbered step less scale on which the intensity increases from left to right, an example can be seen in Figure 28. If a QDA has been done, one should be able to process the obtained data properly. Therefore, the data has to be processed in a statistic way to verify if the results are statistically significant. Often so-called spider-webs are gathered, which can be seen in the Results & Discussion part of this thesis. Those spider-webs show the average of all panellists, the further one point of the spider web is away from the middle of the spider web the more intense the plotted attribute is. The QDA is nowadays, often done with a sensory software so the obtained data is transferred and processed in a detailed manner but can also be done using paper and pen. In this case the CompuSense software was used for the QDA. After the sensory evaluation statistic processing of the obtained data has to be done, e.g. using a statistic tool like XLSTAT as described before [48].

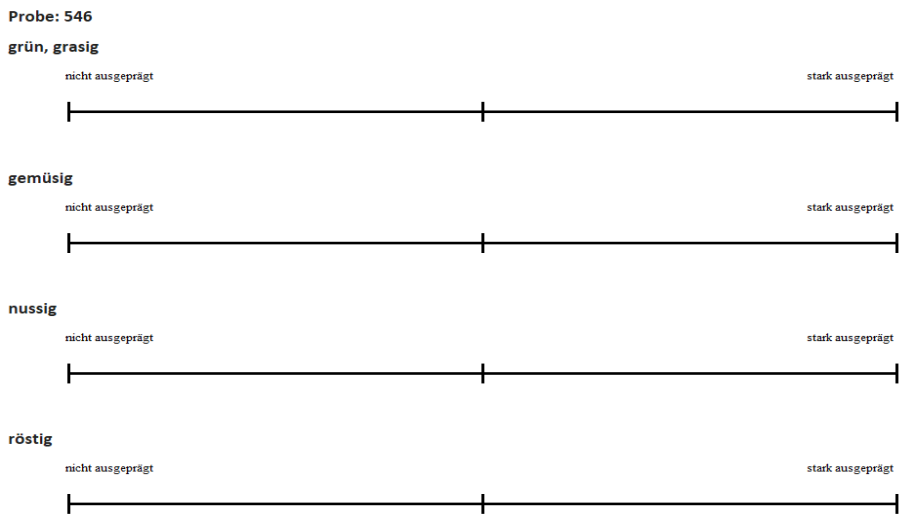


Figure 28: Structure of a typical QDA

The tested samples are coded using three-digit blinding codes to avoid biasing and make sure the samples are evaluated blind. (Figure 29) If analytic sensory evaluation is performed a sensory trained panel has to be used. At Graz University of Technology, the sensory panel is trained following DIN ISO 5492 which states it as a group of individuals that has already performed sensory testing before and meet the requirements of DIN ISO 5492. Additionally, ISO 8586 distinguishes between a testing person and a sensorically trained person. The testing individuals are either laypeople or people who have already taken part in a sensory testing. The first part of ISO 8586 classifies individuals that have been selected to be specially trained in sensory testing and how this training should be done. Part two of this standard gives information about the selection, training and monitoring of so called Expert Sensory Assessors. To sum up ISO 8586, it can be said that, sensorically trained people are selected testers that have been trained and have experience and performance when looking at sensory testing. In contrast ISO 5492:2009 classifies so called experts as individuals that have knowledge or experience in a special field and can position themselves in this field of knowledge [49].

Figure 29: Sample preparation for the sensory testing using QDA

4. Experimental part

The used chemicals, methods, materials and calculations can be found in the following chapter. Additionally, the sample preparation, including the sample sheet and all device parameters will be given. According to the preceding chapter, this chapter will be split into two main parts. At first the material and methods of the evaluation of the rapid test for the determination of fat classification numbers compared to the analytically determination will be listed. The material and methods for the investigation of the roasting, pressing and storage of chia seed, flaxseed and hempseed oil will follow as the second main part.

4.6. Evaluation of a rapid test for the determination of fat classification numbers

4.6.1. Materials and Chemicals

The following chemicals were used for the determination of the fat classification numbers, all chemicals were of the highest commercial grade and used without further purification: ethanol and diethyl ether were purchased from ChemLab NV, Zedelgem, Belgium. Sodium hydroxide solution as well as acetic acid and isooctane were purchased from Carl Roth, Karlsruhe, Germany. Phenolphthalein was obtained from Sigma-Aldrich, Taufkirchen, Germany. Chloroform was purchased from Promochem, Teddington, UK. potassium iodide, starch and sodium thiosulfate solution were obtained from Merck, Darmstadt, Germany. P-anisidine was purchased from VWR, Darmstadt, Germany. The solutions for the evaluated rapid test were supplied by the producer.

4.6.2. Acid number

Ethanol 96 % mixed with diethyl ether in equal parts was used to dissolve the oil sample. Of the oil samples approx. 5 g were weighed and measured in 250 mL glass titration flasks and dissolved with 50 mL of the ethanol and diethyl ether solution. Before titration approx. 4 drops of phenolphthalein were added as an indicator to the mixture. The mix was titrated with 0.1 M sodium hydroxide solution until a colour change to red was seen. Sodium hydroxide solution was used for the titration [36]. The acid number was calculated using Equation 3.

$$AN = \frac{a \cdot N \cdot M}{E} \qquad \textbf{Equation 3}$$

a volume of sodium hydroxide measure solvent used in mL
N titer of the sodium hydroxide solvent in mol/L
E weighed portion of fat in g
M molecular weight of the used alkaline solution

Using the determined acid number, the free fatty acid (FFA) content could be calculated in percent, using Equation 4.

$$FFA = \frac{282 \cdot 100}{40 \cdot 1000} \qquad \textbf{Equation 4}$$

282 molecular weight of oleic acid, used as major fatty acid, in g/mol
40 molecular weight of the used alkaline solution (NaOH) in g/mol

4.6.3. Peroxide value

For the determination of the peroxide value 3 parts of chloroform and 2 parts of 100 % acetic acid were mixed. Furthermore, a 1 % starch solution and a saturated potassium iodide solution was prepared. Approx. 5 g of the oil samples were weighed into 250 mL glass titration flasks. The sample was mixed with 30 mL of the chloroform and acetic acid solution and the potassium iodide was added. The flask was sealed and shook for 60 seconds. After 60 seconds 30 mL of distilled water were added, and the mixture was titrated with 0.01 M sodium thiosulfate solution. When the yellowish colour of the mixture was fading, the starch solution was added giving the mixture a light blue to grey colour. The titration was stopped when no colour was recognizable anymore [36]. The peroxide value is calculated using Equation 5.

$$POZ = \frac{(a-b)\ \cdot\ N}{E}\ \cdot 1000 \qquad \textbf{Equation 5}$$

a usage of sodium thiosulfate solution in the main experiment in mL
b usage of sodium thiosulfate solution in the blind experiment in mL
N concentration of the sodium thiosulfate solution measure solvent in mol/L
E weighed portion of fat in g

4.6.4. Anisidine and Totox value

The anisidine value was determined according to ÖNORM EN ISO 6885:2016. To determine the anisidine value of the oil sample, the photometric measurements of the sample were performed in threefold repetition, every measurement was done in duplicate. To create the anisidine reactant, 0.125 g of solid p-anisidine were weighed into a graduated flask and diluted in 100 % acetic acid. The absorption of the anisidine reactant was then measured against isooctane at 350 nm; as the measured value was lower than 0.2 the solution could be used. As the samples were fluid oils no melting was needed. From every oil 0.5 g of sample were weighed in a 25 mL graduated flask and diluted in isooctane to create the test solution. For the first solution (A_0) that was needed, 5 mL of the test solution was mixed with 1 mL of 100 % acetic acid. For the second solution (A_1) 5 mL of the test solution was mixed with 1 mL of the anisidine reactant. For the third solution, the blind solution, (A_2) 5 mL of isooctane was mixed with 1 mL of anisidine reactant. The created solutions were mixed and were put into darkness. After 8-10 minutes, approx. 1 to 3 mL of the samples were transferred into disposable cuvettes and measured against isooctane at 350 nm [50]. The anisidine value is dimensionless and was calculated Equation 6.

$$AV = \frac{100\ \cdot Q \cdot V \cdot [1.2 \cdot (A_1 - A_2 - A_0)]}{m} \qquad \textbf{Equation 6}$$

V volume the sample was diluted in in mL
m mass of the oil sample in g
Q sample content of the measured solution the anisidine number was given in g/L
A_0 extinction of the test solution before the reaction
A_1 extinction of the test solution after the reaction
A_2 extinction of the blind solution
1.2 correction factor of the dilution of the test solution

The Totox value was calculated using Equation 7 for all samples.

$$TV = (2 \cdot PV) + AV \qquad \textbf{Equation 7}$$

PV peroxide value
AV anisidine value

4.7. Investigation of the volatile compounds from roasting, pressing and storage process of selected oils

4.7.1. Materials and Chemicals

The following chemicals were used for the investigation of the volatile compounds, all chemicals were of the highest commercial grade and used without further purification: 2-pentanol, methanolic boron trifluoride, natriumchloride, FAME standard as well as dichlormethane were obtained from Sigma-Aldrich, Taufkirchen, Germany. Methanol was purchased from VWR, Darmstadt, Germany. Helium was purchased from Linde, Stadl-Paura, Austria. Triundecanoine was purchased from Larodan, Solna, Sweden and heptane was obtained from VWR, Darmstadt, Germany.

4.7.2. Sampling

Ready to use oil samples were provided by oil mill Fandler. Hempseed oil, flaxseed and chia seed oil samples with different BBD were taken. The sample table can be seen in Table 4. The samples of the roasting and pressing process of the oils were directly taken at the oil mill Fandler. Therefore, samples were taken of the raw material, the squeezed seeds and after the roasting process was started, every 5 minutes directly from the heat pan. Surface temperature was recorded using an infra-red thermometer that allows hygienic measurement without food contact. (TLC 720, Ebro, Weilheim, Germany) on the surface of the seeds in the heating pan. Figure 30 shows the roasting temperature curves of all investigated oil seeds. The hempseeds are roasted for approx. 40 minutes, the chia and flaxseeds are roasted for around 35 minutes. This value can vary with other raw material, as the roasting process is an individual process for every new raw material. The temperature was pre-set for the bottom of the heat pan. For the chia seeds the temperature was set to 110°C, for flaxseed to 105°C and for the hempseeds to 183°C. It is important to mention here, that previously performed heat measurements at the oil mill Fandler have shown that the set temperatures can differ from the temperature that the heat plate really reaches. The higher the temperature the greater the deviation from the set temperature. That being said, it is possible that the temperature is set to 180°C but the temperature experienced at the heat plate is at approx. 120-130°C.

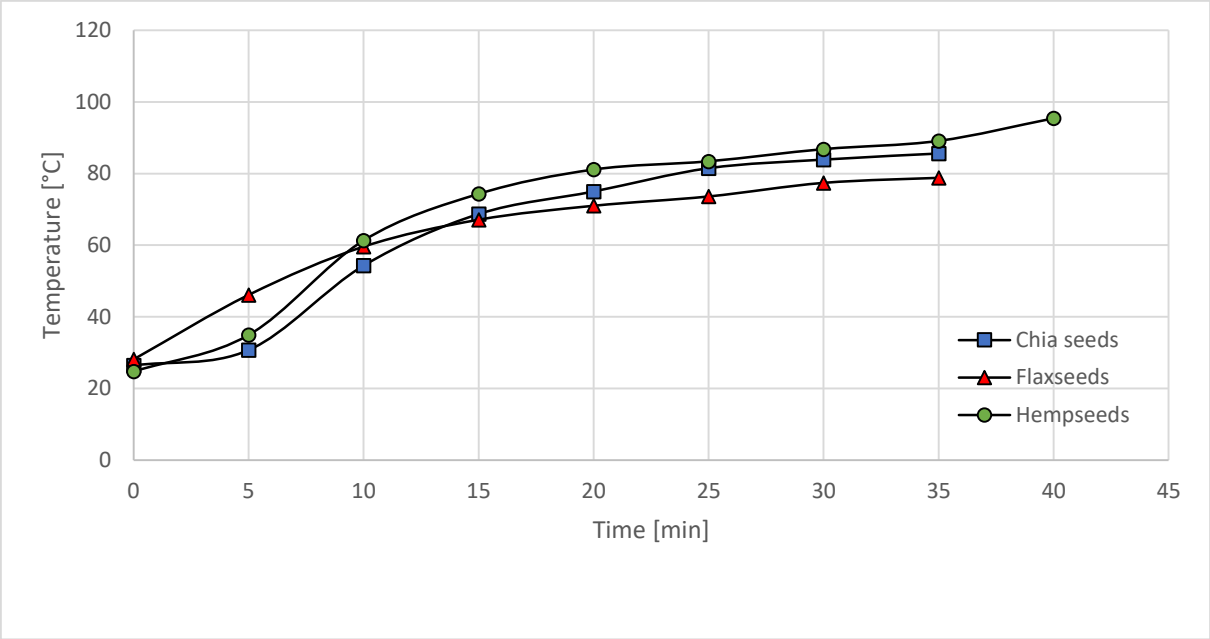


Figure 30: Course of surface temperature for all three roasting processes, measured with an infrared thermometer on the surface of the seeds in the heat pan

Approx. 4 L of water are added to approx. 50-60 kg of the hempseeds at the beginning of every roasting. The water is added to the seeds to prolong the roasting time and give the seeds more time to dry and develop their typical flavour. If the water was not added to the seeds for the roasting process, the seeds would brown and roast too fast and thereby cause a burnt flavour. Furthermore, the yield of oil from the seeds can differ significantly. From the pressing of around 50-60 kg of chia seeds around 13 L of oil are gained. In contrast, the pressing of 50-60 kg of hempseeds will yield around 25 L of oil. Samples that were used for the analysis of the volatile compounds, were directly taken from the roasting pan and put into 30 mL vials with a screw cab. The taken samples were labelled and were put on ice immediately after sampling. After roasting of the chia seed and hempseed oil samples were taken directly from the press, one day after pressing, after the storage period in the tanks and from the freshly filled oil. From the flaxseed oil, samples were taken directly after pressing and from the filled oil, because the flaxseed oil has only a storage period of one day prior to filling. The samples directly taken from the pressing of the seeds were taken in 30 mL vials with a screw lid and put on ice. Oil samples were directly filled into dark coloured glass bottles and delivered to the lab. Samples from the roasting process were deep frozen until further use, oil samples in glass bottles were stored in the cooling room in the dark at approx. 4°C.

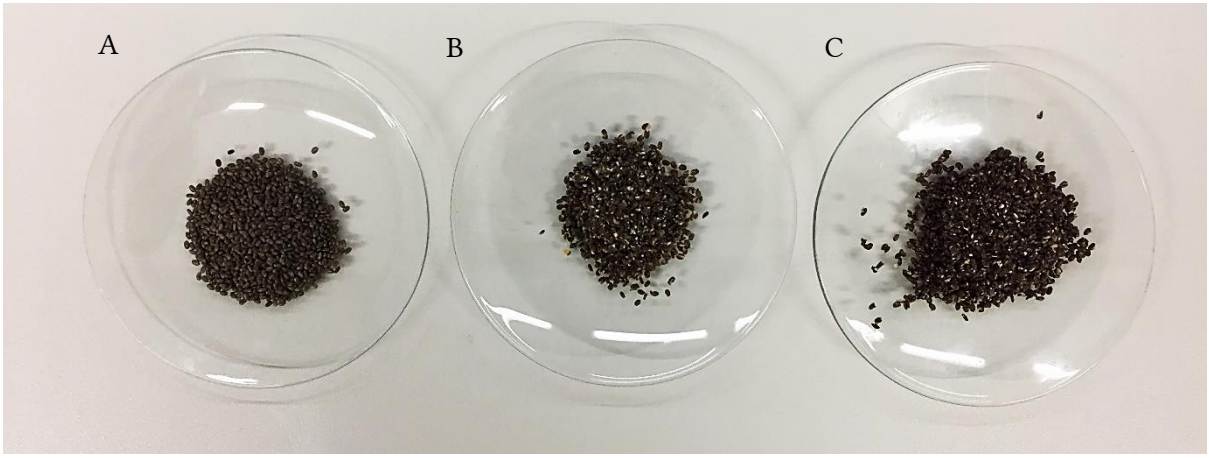


Figure 31: Roasting process of the chia seeds, A = Raw material, B = Squeezed seeds, C = 30 Minutes of roasting

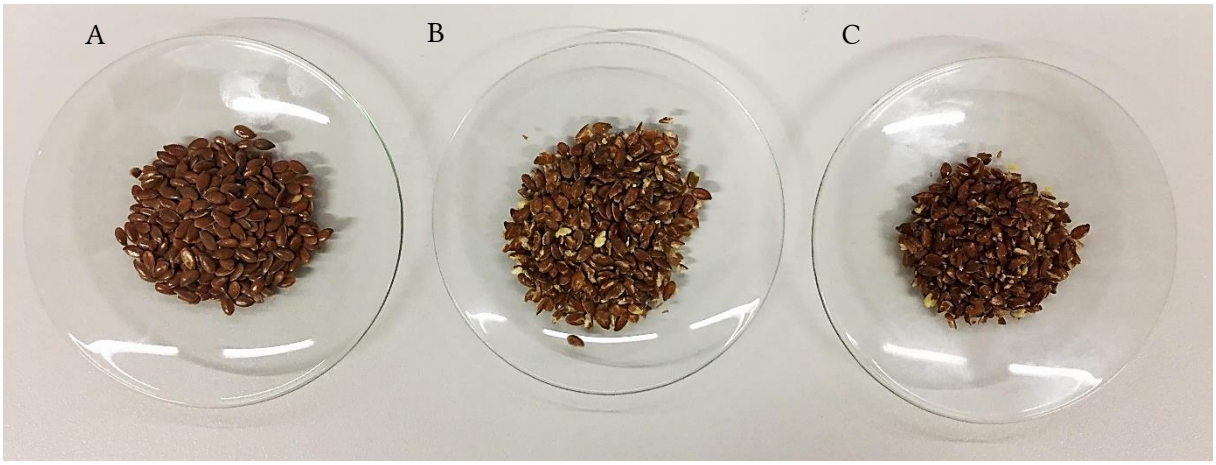


Figure 33: Roasting process of the flaxseeds, A = Raw material, B = Squeezed seeds, C = 30 Minutes of roasting

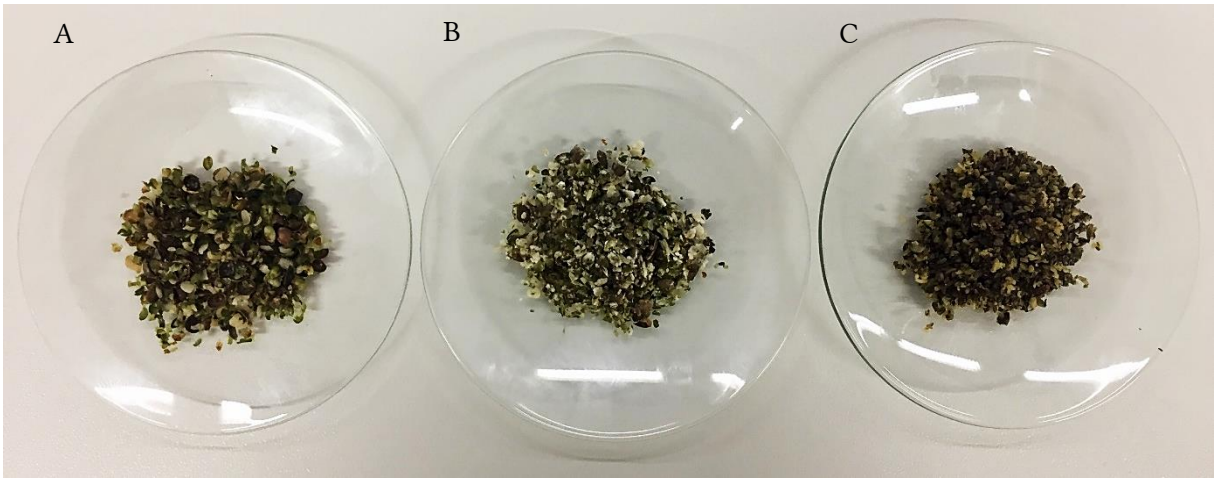


Figure 32: Roasting process of the hempseeds, A = Raw material, B = Squeezed seeds, C = 30 Minutes of roasting

Table 4: Overview of all bottled oil samples investigated in the thesis including the analysis dates

Sample	Lot	Size [mL]	Production date	BBD	Storage conditions	Acid number	Peroxide value	Anisidine value	SPME-GCMS	Fatty acids	Sensory evaluation
Organic flaxseed oil	C170197	100	09.02.2017	27.05.2017	cool and dry place	10.07.2017	11.07.2017	18.08.2017	20.06.2017	27.06.2017	
Flaxseed oil	C170049	100	10.02.2017	27.05.2017	cool and dry place	10.07.2017	11.07.2017	18.08.2017	20.06.2017	27.06.2017	
Organic flaxseed oil	C170211	250	18.05.2017	05.09.2017	cool and dry place	10.07.2017	11.07.2017	18.08.2017	20.06.2017	27.06.2017	12.07.2017
Flaxseed oil	C170061	250	19.05.2017	29.08.2017	cool and dry place	10.07.2017	11.07.2017	18.08.2017	20.06.2017	27.06.2017	12.07.2017
Organic chia seed oil	D160207	100	22.01.2016	01.03.2017	cool and dry place	10.07.2017	11.07.2017	18.08.2017	20.06.2017	21.06.2017	
Organic chia seed oil	C160182	100	17.11.2016	28.11.2017	cool and dry place	10.07.2017	11.07.2017	18.08.2017	20.06.2017	21.06.2017	12.07.2017
Organic chia seed oil	C170134	100	07.04.2017	15.05.2018	cool and dry place	10.07.2017	11.07.2017	18.08.2017	20.06.2017	21.06.2017	12.07.2017
Organic hempseed oil	C160204	100	22.03.2016	01.04.2017	at room temperature	10.07.2017	11.07.2017	18.08.2017	20.06.2017	27.06.2017	
Hempseed oil	C150019	100	09.12.2015	01.04.2017	at room temperature	10.07.2017	11.07.2017	13.07.2017	20.06.2017	27.06.2017	
Organic hempseed oil	C160209	100	09.09.2016	03.11.2017	at room temperature	10.07.2017	11.07.2017	18.08.2017	20.06.2017	27.06.2017	
Hempseed oil	C160024	100	18.08.2016	27.10.2017	at room temperature	10.07.2017	11.07.2017	13.07.2017	20.06.2017	27.06.2017	
Organic hempseed oil	C170153	250	06.04.2016	03.05.2018	at room temperature	10.07.2017	11.07.2017	18.08.2017	20.06.2017	27.06.2017	12.07.2017
Hempseed oil	C160026	250	02.11.2016	03.05.2018	at room temperature	10.07.2017	11.07.2017	13.07.2017	20.06.2017	27.06.2017	

4.7.3. Headspace SPME GC-MS

For the analysis of the volatile compounds of all samples, gas chromatography mass spectrometry was used. For the enrichment of the volatiles headspace solid phase micro extraction (HS-SPME) was used. From every point of sample taking four measurements were done. Prior to the measurements the samples were sealed with a cap. For the HS-SPME extraction a Supelco 50/30 µm DVB/Carboxen/PDMS Stable Flex Fibre with a length of 2 cm (Merck, Darmstadt, Germany) was used with an extraction time of 10 minutes and an extraction temperature of 40°C for the fibre. From the roasted seeds and stored oil approx. 200 mg were weighed into 20 mL headspace vials with a glass coated magnetic stirrer. In those samples, an internal standard was used additionally. As internal standard 2-pentanol diluted in MeOH was used with a concentration of 10 mg/L, to every sample 10 µl of the standard was added, giving an absolute concentration of 100 ng in the sample itself. With every measurement, a SPME mix as well as a mix of n-alkanes was analysed, the SPME mix is measured to pursue the performance of the system, the mixture of n-alkanes is needed to calculate the retention indices in the course of identifying the compounds. (Table 5 and 6) For both 10 µL from a 1 mg/L in methanol (MeOH) standard was used, giving a concentration of 10 ng in the vial. After the measurements were done at the HS-SPME GC-MS the data sets were evaluated with the PARADISe software of the University of Copenhagen [46]. From the gained results, the average of the peak area was calculated (n=4), as well as the concentration relative to the internal standard and in combination with available odour thresholds the OAV were calculated. The average of the peak areas for all obtained samples were processed using XLSTAT. Measurement parameters for the samples and n-alkane mixture can be found in Table 7, parameters for the SPME mix can be found in Table 8.

Table 5: Composition of the used SPME mix

Compound	Concentration [g/L]
Heptanal	1.364 l
α-Pinene	1.016
β-Pinene	0.976
Decane	0.966
Octanal	0.945
p-Cymene	1.090
1,8-Cineol	1.015
Nonanal	1.078
Menthol	1.040
Dodecane	1.034
t-Carveol	2.006
Carvone	1.180
Decanol	0.972
Undecanol	0.966
Tetradecane	0.983
Dodecanol	0.960

Table 6: Composition of the used n-alkane mixture

Compound
α-Pinene
β-Pinene
p-Cymene
1,8-Cineol
L-Menthol
1-Dodecanol
Methyldecanoate
Acenaphthene
n-Alkane (C8-C20)

Table 7: GC-MS parameters for the measurement of the samples of the roasting, pressing and storage process as well as for the bottled oils and the measured n-alkane mixture

Device parameters	
Gas chromatograph	Agilent Technologies 7890A GC
Detector	MSD, Triple-Axis Detector, EI
Column parameters	
Column	HP-5
Column length	27 m
Column inner diameter	250 µm
Film thickness	0.25 µm
Carrier gas	Helium
Method parameters	
Injector temperature	270°C
Detector temperature	260°C
Injection mode	Splitless
Mode	Constant flow
Measurement mode	scan
Mass range	20-300 amu
Flow rate (constant flow)	33 cm/sec
Solvent delay	4 min
Temperature program	-10°C (1min) – 8°C/min – 260°C

Table 8: GC-MS parameters for the measurement of the samples of the SPME mix measured with every roasting, pressing and storage process as well as for the bottled oil samples

Device parameters	
Gas chromatograph	Agilent Technologies 7890A GC
Detector	MSD, Triple-Axis Detector, EI
Column parameters	
Column	HP-5
Column length	27 sm
Column inner diameter	250 µm
Film thickness	0.25 µm
Carrier gas	Helium
Method parameters	
Injector temperature	270°C
Detector temperature	260°C
Injection mode	Splitless
Mode	Constant flow
Measurement mode	scan
Mass range	35-300 amu
Flow rate (constant flow)	32 cm/sec
Solvent delay	5 min
Temperature program	35°C (1min) – 5.5°C/min – 230°C

4.7.4. Sensory evaluation

Two different sensory methods were used to evaluate the sensory characteristics of the flaxseed, chia seed and hempseed oil, using a sensory expert panel. The expert panel of Graz University of Technology consists of individuals trained on the handling of different analytical sensory problems. For the oil samples no, specific training was needed for working on the described problems. For the descriptive evaluation, two samples of each oil were given to the test panel. No specific vocabulary had to be trained for the descriptive testing. The panellists evaluated the samples in a separate booth, one sample at a time in randomized order. The samples were coded with a random three-digit code. The panellists entered their scoring into a tablet computer. All tested oil samples were in their durability period as the testing was done on the 12.07.2017. For hempseed and flaxseed oil organically and conventionally produced oil samples were chosen. As the chia seed oil is only produced as organic chia seed oil two organic chia seed oil samples were given to the panel, as seen in Table 9. The information was given that the panel will test flaxseed, chia seed and hempseed oil, as well as if the samples are organic produce or not. The oils were coded with three-digit number codes and the panellists were told which kind of oil they are testing. As the panellists were asked to also describe the colour of the oils, clear glass cups were used for the oil testing. 5 mL of every oil were given in the cups and covered with aluminium caps prior to testing. The evaluation sheet (written in German) can be found in the Appendix.

Table 9: Sample sheet of the investigated oils

Type of oil	Best before date
Organic chia seed oil	28.11.2017
Organic chia seed oil	15.05.2018
Organic flaxseed oil	05.09.2017
Flaxseed oil	29.08.2017
Organic hempseed oil	03.05.2018
Hempseed oil	03.05.2018

The first evaluation was mainly done to make the panellists familiar with the different properties of the oils. Therefore, on the test sheets short descriptions of the characteristics of the different tested oils were given and could be used from the panellists to describe the samples. Additionally, to the sensory evaluation of the oil samples themselves, also sniffing sticks were tested by the expert panel at another testing session. The selection of the compounds that were used for the sniffing sticks was based on the results obtained from GC-MS analysis. The sticks were dipped into ethanolic dilutions of the different compounds and left to dry for a couple of minutes. The sticks were prepared approx. 1 hour prior to the sensory evaluation. The panel was given a test sheet were the odour impression should be described. Also, the table of the chosen compounds can be found in the Appendix. For the second sensory technique, the sensory expert panel of Graz University of Technology was asked to perform quantitative-descriptive analysis (QDA) of hempseed oil samples. CompuSense Sensory Software (CompuSense, Guelph, Canada) was used for the data acquisition and statistical evaluation of the results. The used attributes were chosen according to the odour characteristic and intensity of the found volatile compounds in hempseeds and hempseed oil. For the first QDA, 2 g of each sample were weighed into blue coloured oil glasses and covered with aluminium lids. As samples the raw hempseeds, hempseeds after 10 minutes of roasting, after 20 minutes of roasting, after 30 minutes of roasting, after 40 minutes of roasting and the hempseed oil directly after pressing was used. The samples were given to the panellists in randomized order with three-digit number codes. The given samples should be ranked on an unnumbered scale in the attributes of nutty, roasted, green/grassy and vegetable-like odour. For the second evaluation, also 2 g of the sample were weighed into blue coloured oil glasses and covered with aluminium lids. Hempseed oil directly after pressing, oil after one day of pressing, oil after the storage in a larger tank and oil after filling in the oil bottle were used. The samples were given to the panellists in randomized order with three-digit number codes. The given samples should be ranked on an unnumbered sale in the attributes of nutty, roasted, green/grassy, rancid and vegetable-like odour.

4.7.5. Investigation of the fatty acid composition

4.7.5.1. Sample preparation

To be able to analyse the fatty acid composition, the glycerides had to be transferred in to the corresponding fatty acid methyl esters (FAME). For the transesterification 20 mg were weighed in 50 mL vials, the measurements were performed in duplicates. 35 µL of internal standard (30.12 g/L triundecanoine dissolved in heptane) was added. 6 mL 0.5 M methanolic NaOH was added to the sample mixed with the internal standard. A Teflon-coated magnetic stirrer was added, and the samples were mixed for 30 minutes at 80°C in a heating block while stirring. After 30 minutes, the samples were cooled to room temperature and 4.5 mL of 20% methanolic boron trifluoride was added. The samples were again placed in a heating block and left there for 15 minutes at 80°C while stirring. Again, the samples were cooled to room temperature and the magnetic stirrers were removed from the sample vials. To extract the fatty acid methyl esters 10 mL of saturated, aqueous NaCl solution and 10 mL heptane were added to the samples.

The samples were mixed for 1 minute on the vortex (mixer/shaker). An aliquot of the upper phase (heptane phase) was filled into micro vials and was analysed by the GC-FID.

4.7.5.2. Measurements

With the GC-MS analysis of FAME (Fatty Acid Methyl Esters) samples a vial with heptane alone was added, as well as a vial with the internal standard, a vial with the derivation reactants was added and a FAME standard.

The FAME standard used was a Supelco 37 Component FAME 10 g/L dissolved in dichlormethane with a final concentration of 2 g/L in heptane, 1 µL was injected. From every oil two vials with oil samples were prepared and those samples were measured twice at the GC. Measurement parameters can be found in Table 10.

Table 10: GC-FID parameters for the determination of the fatty acid composition from the bottled oils

Device parameters	
Gas chromatograph	GC HP 5890 Series II
Detector	FID
Column parameters	
Column	ZB-WAX
Column length	30 m
Column inner diameter	0,32 mm
Film thickness	0,25 µm
Carrier gas	Helium
Method parameters	
Injector temperature	250°C
Detector temperature	260°C
Injection mode	Split injection, Split ratio 1:13
Mode	Constant flow
Inlet pressure	145 kPa
Flow rate	30 mL/min
Temperature program	50°C/1min – 15°C/min – 140°C –8°C/min – 250°C

5. Results and Discussion

The following chapter will deal with the results obtained within this study and discuss those. For the first part all determined fat classification numbers will be given, compared to the results obtained using the evaluated rapid test. These results will be discussed and compared to values found in literature. The second main part of the study will follow with the determined volatile compounds of the roasting and pressing process. Statistic evaluation and graphic representation of the obtained data is given as well. Followed by discussion and comparison of these values with literature. The sensory evaluation of the samples can also be found in this chapter.

5.1. Evaluation of a rapid test for the determination of fat classification numbers

Figure 34 shows a comparison of the acid number, peroxide value, anisidine and Totox value for all investigated bottled oils. As can be seen from Table 11 the determination of the acid number was performed in duplicates, as well as the peroxide and anisidine value given in Table 12 and Table 13. Table 14 shows the calculated Totox values for the evaluated oils. The corresponding data obtained from the cdR FoodLab junior rapid test system is shown in Table 15.

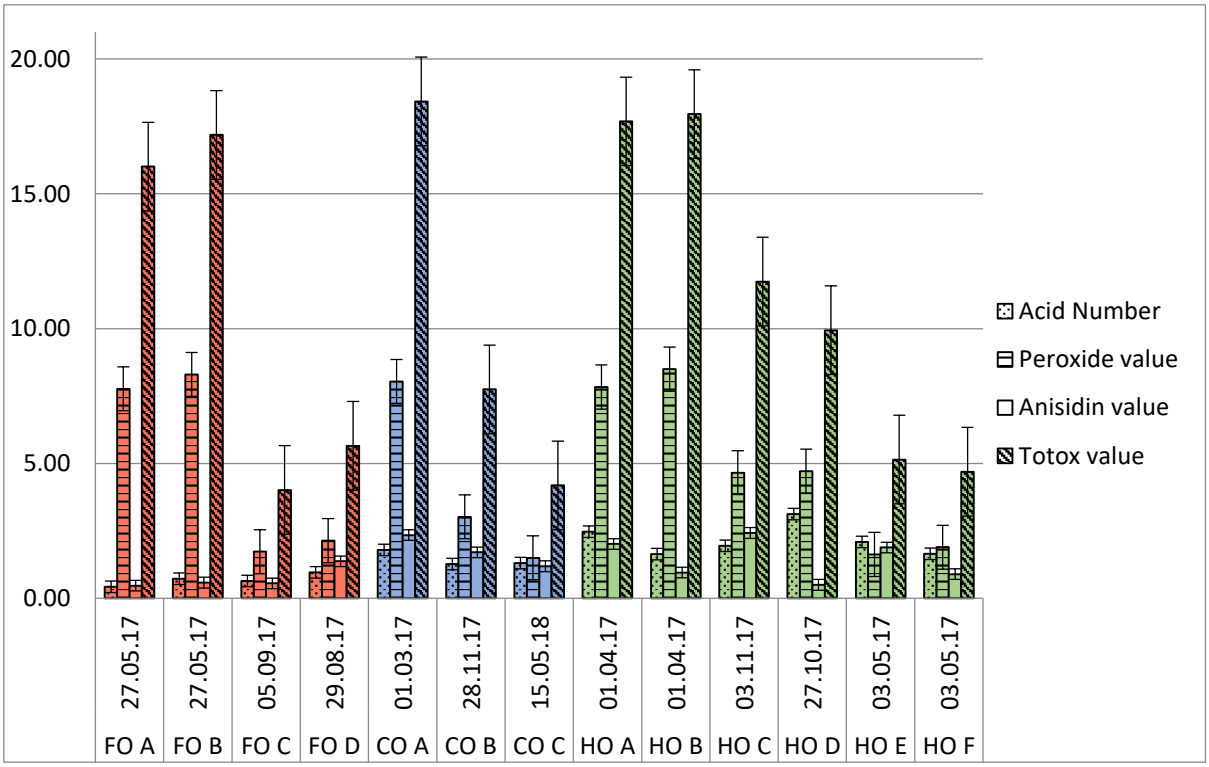


Figure 34: Comparison of the acid number, peroxide value, anisidine value and Totox value of the investigated oils, the best before date for all oils is given, FO = Flaxseed oil, CO = Chia seed oil, HO = Hempseed oil

5.1.1. Acid number

Table 11: Acid numbers of the investigated oils, FFA in %

Sample	BBD	Acid number 1	Acid number 2	Average	FFA [%]
Organic flaxseed oil	27.05.2017	0.42	0.43	0.43	0.30
Flaxseed oil	27.05.2017	0.75	0.70	0.73	0.51
Organic flaxseed oil	05.09.2017	0.67	0.61	0.64	0.45
Flaxseed oil	29.08.2017	0.93	1.01	0.97	0.68
Organic chia seed oil	01.03.2017	1.78	1.81	1.79	1.26
Organic chia seed oil	28.11.2017	1.25	1.30	1.27	0.90
Organic chia seed oil	15.05.2018	1.32	1.31	1.31	0.93
Organic hempseed oil	01.04.2017	2.45	2.50	2.47	1.74
Hempseed oil	01.04.2017	1.63	1.65	1.64	1.16
Organic hempseed oil	03.11.2017	1.91	1.99	1.95	1.38
Hempseed oil	27.10.2017	3.13	3.12	3.13	2.20
Organic hempseed oil	03.05.2017	2.09	2.10	2.09	1.47
Hempseed oil	03.05.2017	1.66	1.65	1.65	1.17

The determined acid number of the investigated **flaxseed oils** ranged from 0.43-0.97. A variation of 0.3 between the organic flaxseed oil and the commercial flaxseed oil could be determined. Krist *et al.* [8] reported an acid number for flaxseed oil of < 4.0. Looking at the acid number of the flaxseed oil samples, no increase or decrease depending on the BBD can be seen. As mentioned above small deviations between the commercial and the organic flaxseed oil can be seen which could depend on the different raw material. Different storage conditions before pressing the seeds and after the pressing could affect the acid number and therefore the content of free acids in the oil. However, all of these values still range under the given literature value of < 4.0 [8]. The *et al.* [51], reported in 2013 a FFA for flaxseed oil of 0.75, which correlates with the FFA determined in this study.

The determined acid numbers for the **chia seed oil** were noticeably higher as they ranged from 1.79-1.31. Particularly, the acid number from the sample with a BBD 01.03.2017 was significantly higher. The chia seed oil samples show a slight decrease with an increasing BBD. These differences are only ± 0.5, which can be referred to a different raw material used for the three different oils as well as to different storage conditions before and after the pressing. Krist *et al.* [8] reported, an acid value of approx. 2.0 is found for chia seed oil. For all chia seed oil samples an acid number below the literature value was determined. Imran *et al.* [52] showed that the storage conditions of raw chia seed oil can have an impact on the FFA. If the chia seed oil is stored for 30 days at 25°C the FFA was reported with 1.7 but stored for the same period of time at 4°C the FFA was 1.32. With increasing storage up to 60 days the FFA at 25°C increased to 2.2., at 4°C to 1.65 [52].

The acid values for the **hempseed oil** samples varied between 1.65-3.13. Looking at the hempseed oil samples bigger differences in the determined acid numbers between organic and commercial hempseed oil can be seen. Acid numbers differ ± 1.0 between commercial an organic hempseed oil with the same BBD. As mentioned already for flaxseed and chia seed oil these differences can be referred to a different raw material. Additionally, different storage conditions of the seeds, the oil tanks and the filled oils could affect the acid number. Krist *et al.* [8] reported an acid number of 3.98 for hempseed oil. The determined acid numbers for all hempseed oil samples did not exceed the given literature value [8]. In 2013 The *et.al* [51], reported a FFA for hempseed oil of 0.89, compared to the investigated hempseed oil samples in this study the determined values are elevated.

5.1.2. Peroxide value

Table 12: Peroxide value of the tested oils

Sample	BBD	Peroxide value 1	Peroxide value 2	Average
Organic flaxseed oil	27.05.2017	7.75	7.80	7.77
Flaxseed oil	27.05.2017	8.27	8.33	8.30
Organic flaxseed oil	05.09.2017	1.77	1.68	1.73
Flaxseed oil	29.08.2017	2.08	2.19	2.14
Organic chia seed oil	01.03.2017	7.92	8.16	8.04
Organic chia seed oil	28.11.2017	2.88	3.16	3.02
Organic chia seed oil	15.05.2018	1.37	1.63	1.50
Organic hempseed oil	01.04.2017	7.64	8.03	7.84
Hempseed oil	01.04.2017	8.56	8.44	8.50
Organic hempseed oil	03.11.2017	4.11	5.22	4.66
Hempseed oil	27.10.2017	4.94	4.50	4.72
Organic hempseed oil	03.05.2017	1.71	1.55	1.63
Hempseed oil	03.05.2017	1.88	1.92	1.90

The peroxide value for the two investigated **flaxseed oils** that already exceeded their BBD at the time of analysis (11.07.2017) were determined with 7.8 and 8.3. For the two oils that were still before their BBD (05.09.2017 & 29.08.2017) at the time of the analysis the peroxide value was noticeable lower (at approx. 2.0). The progression of the peroxide value was expected as the peroxide value increases with aging of the oil. No noticeable differences between the commercial and the organic flaxseed oil can be detected. All determined peroxide values for the flaxseed oil samples are under the given guidelines of the Austrian Codex Alimentarius, which describes that the peroxide value should be lower than 10.0 for unrefined oils [25]. In 2007 Choo *et al.* [53], tested seven different cold-pressed flaxseed oils from New Zealand, all of which had a peroxide value under the given regulations with a peroxide value of less than 10.0.

Quite similar progression could be seen in the three **chia seed oil** samples, as the peroxide value from the oil with the longest BBD with 1.5 in comparison to the oil sample with the shortest BBD with a detected value of 8.04 was obtained. The peroxide values show an expected progression over the storage time, as the peroxide value increases according to a longer storage period. However, none of the determined values exceeds the guidelines of a peroxide value of lower than 10.0 given by the Austrian Codex Alimentarius [25]. Ixtaina *et al.* [54], reported a peroxide value of 1.0 for the chia seed oil from Argentina. Imran *et al.* [52] showed in 2016 peroxide values ranging from 0.67-2.67 for raw chia seed oil. The peroxide values for the samples still within their BBD were found to be in the same range.

This trend is also seen in the **hempseed oil** samples, as in these samples the peroxide value of the still durable oils is detected around 5.0-1.5 in contrast to the exceeded oil samples with a value of approx. 8.0. The samples show an expected progression over the time as the peroxide value increases with an increasing BBD and a prolonged storage period. As already mentioned for the flaxseed oil samples, also the hempseed oil samples show no significant difference between commercial and organic produced oil. The Austrian Codex Alimentarius also gives a maximum peroxide value for unrefined oils of lower than 10.0, which is not exceed by the determined values for the hempseed oil samples [25]. In 2013 Teh *et.al* [51]., determined a peroxide value of 1.94 for hempseed oil.

5.1.3. Anisidine value

Table 13: Anisidine values of the tested oils, AV = average of the gathered values, AN = calculated anisidine value

Sample	BBD	A0	A0	AV	A1	A1	AV	A2	A2	AV	AN
Organic flaxseed oil	27.05.2017	0.07	0.07	0.07	0.14	0.14	0.14	0.04	0.04	0.04	0.48
Flaxseed oil	27.05.2017	0.06	0.06	0.06	0.14	0.14	0.14	0.04	0.04	0.04	0.58
Organic flaxseed oil	05.09.2017	0.09	0.08	0.08	0.16	0.17	0.16	0.05	0.04	0.04	0.56
Flaxseed oil	29.08.2017	0.08	0.08	0.08	0.52	0.51	0.52	0.04	0.05	0.04	5.88
Organic chia seed oil	01.03.2017	0.09	0.09	0.09	0.31	0.27	0.29	0.05	0.04	0.05	2.34
Organic chia seed oil	28.11.2017	0.09	0.09	0.09	0.26	0.25	0.25	0.05	0.05	0.05	1.71
Organic chia seed oil	15.05.2018	0.09	0.10	0.10	0.21	0.22	0.22	0.04	0.04	0.04	1.19
Organic hempseed oil	01.04.2017	0.01	0.10	0.05	0.19	0.19	0.19	0.06	0.05	0.05	2.27
Hempseed oil	01.04.2017	0.11	0.12	0.11	0.25	0.23	0.24	0.07	0.05	0.06	3.82
Organic hempseed oil	03.11.2017	0.09	0.11	0.10	0.21	0.17	0.19	0.05	0.05	0.05	2.44
Hempseed oil	27.10.2017	0.13	0.13	0.13	0.22	0.21	0.21	0.05	0.05	0.05	2.00
Organic hempseed oil	03.05.2017	0.10	0.09	0.10	0.16	0.19	0.17	0.04	0.04	0.04	1.91
Hempseed oil	03.05.2017	0.09	0.09	0.09	0.19	0.21	0.20	0.06	0.03	0.05	3.59

The anisidine values for the **flaxseed oils** that exceeded their BBD at the time of the analysis (18.08.2017) showed a value between 0.1 – 0.5. Between the commercial and the organic samples with the BBD 27.05.2017 no significant difference can be seen. However, the commercial flaxseed oil with a BBD until 29.08.2017 showed a significantly higher anisidine value with 5.88 compared to the other samples. Different raw material or different storage conditions of the raw material or the pressed oil could have influenced this elevated value. As the anisidine value describes the α, β-unsaturated aldehydes and therefore gives an idea of the history of an oil samples and its durability. The oil sample could have already exceeded its particular durability range. Choo *et. al.* [53], reported an anisidine value of less than two for a good quality oil. Expect the flaxseed oil with a BBD until 29.08.2017. all other oils are within this range.

The **chia seed oil** values are found to be 2.0 for the exceeded sample and around 1.0 in the still durable sample. As expected the values for the already exceed sample is higher compared to the two samples still in the range of their BBD. Ixtaina *et al.* [54], found anisidine values of 1.7 after 225 days of storage at 4°C were found for chia seed oil.

The **hempseed oil** samples showed an anisidine value at around 4.0 for the exceeded samples of the oil and of around 2.0 for still durable samples. Between the commercial and the organic samples differences could be found. This could be depended on the different raw material and the different storage conditions after pressing. Also, the age and storage conditions of the raw material could influence the differences. Anisidine values starting at 0.9 (±0.57) at 0 months of storage increasing to 1.09 (±0.73) after 6 months of storage, were found by Perscha *et. al.* [55], for different cold pressed oils.

5.1.4. Totox value

Table 14: Calculated Totox value using the gathered peroxide and anisidine value of the tested oils

Sample	BBD	Totox value
Organic flaxseed oil	27.05.2017	16.02
Flaxseed oil	27.05.2017	17.18
Organic flaxseed oil	05.09.2017	4.01
Flaxseed oil	29.08.2017	10.15
Organic chia seed oil	01.03.2017	18.42
Organic chia seed oil	28.11.2017	7.75
Organic chia seed oil	15.05.2018	4.20
Organic hempseed oil	01.04.2017	17.94
Hempseed oil	01.04.2017	20.82
Organic hempseed oil	03.11.2017	11.76
Hempseed oil	27.10.2017	11.44
Organic hempseed oil	03.05.2017	6.21
Hempseed oil	03.05.2017	7.39

The Totox value was calculated from the determined peroxide and anisidine values as described in 4.1.3. The Totox value should give an idea of the overall oxidation of a certain fat or oil, however the value has only orientating character.

The Totox values of the **flaxseed oils** ranges from approx. 17.0 to around 4.0 as the BBD prolongs. The Federal Ministry of Food and Agriculture Germany defined values for cooking oil, where it is stated that the Totox value should not exceed the value of 20 for cold pressed and native oils [56]. All determined values for the flax seed oil samples remain under this given value. As the Totox value is calculated using the determined peroxide and anisidine value, the flaxseed oil with the BBD 29.08.2017 has an elevated value, as expected considering the elevated anisidine value. However, between the organic and commercial flaxseed oil sample with the same BBD (27.05.2017) no significant differences could be determined. Choo *et al.* [53] reported a Totox value of under 4 for good quality oil. Only the organic flaxseed oil with BBD of 05.09.2017 is within this range. Choo *et al.* [53] tested 7 different cold-pressed flax seeds oils from New Zealand, 4 of these oils showed a Totox value under 4.

The values for the **chia seed oil** samples show an expected progression over time, as the values decrease with a longer BBD. Ixtaina *et al.* [54], reported a Totox value of 21.5 for chia seed oil that was stored for 225 days at approx. 4°C. It should be mentioned that the storage of chia seed oil can have a great impact on the oxidative parameters of the oil. As shown by Ixtaina *et al.* [54], in 2011 chia seed oil that was stored for the same period at 4°C showed significantly lower values in anisidine value, peroxide value and Totox value compared to the oil stored at 20°C. The chia seed oil samples used in these investigations was always stored at room temperature according to the recommendations of the producing company.

The Totox values for the investigated **hempseed oil** samples range from around 18.0 to 4.0 respectively from 21.0 to around 6.0-7.0. No significant differences could be determined in the commercial and organic oil samples. As the Federal Ministry of Food and Agriculture Germany defines a Totox value of under 20.0, the samples are under this given range, except the commercial hempseed oil with the BBD 01.04.2017, which exceeds this value [56].

5.1.5. Results obtained by the cdR FoodLab Junior rapid test system

Table 15: Acid numbers, peroxide values and anisidine values obtained using the provided test kits from cdR, peroxide value is given in meqO_2/kg, anisidine value is given in AnV; time of the analyses

Sample	BBD	Acid number [%]	Peroxide value	Anisidine value
Organic flaxseed oil	27.05.2017	0.49	0.67	0.5
Flaxseed oil	27.05.2017	0.90	0.45	0.7
Organic flaxseed oil	05.09.2017	0.77	<0.30	0.6
Flaxseed oil	29.08.2017	0.78	0.46	0.7
Organic chia seed oil	01.03.2017	3.40	8.32	1.3
Organic chia seed oil	28.11.2017	0.88	4.48	0.7
Organic chia seed oil	15.05.2018	1.00	2.11	0.7
Organic hempseed oil	01.04.2017	2.70	5.09	1.5
Hempseed oil	01.04.2017	1.80	2.27	1.4
Organic hempseed oil	03.11.2017	2.70	4.5	1.5
Hempseed oil	27.10.2017	2.50	1.34	1.4
Organic hempseed oil	03.05.2017	0.78	4.52	1.4
Hempseed oil	03.05.2017	1.20	4.94	0.6

The results obtained for the acid numbers with the rapid test system cdR Food Lab Junior showed the same tendency as those values that were obtained by the traditional volumetric technique. The highest differences were found for the peroxide and the anisidine value as seen in Table 15. Significant differences were found in the results for flax and hempseed oil. On the one hand, the peroxide values for chia seed oil determined with the rapid test Food Lab Junior matched with the results from the volumetric determination. In contrast, the results for the peroxide values for flaxseed oil showed large deviations between the two methods. Due to the high differences in the results, we aimed to identify possible reasons for the divergence of the obtained results. The first idea was to verify the influence of UV absorption of the oils on the method. This was considered to be of interest especially for hempseed oil due to its colour and the reported concentrations of tocopherols, carotenoids and chlorophylls. According to the information of the supplier of the rapid test, the photometric determination was performed at 505 nm for the determination of the peroxide value. To evaluate a possible interference by oil constituents, the pure oils were analysed at 505 nm and the UV VIS spectrum was determined. Furthermore, the oils were diluted with hexane according to the dilution that is used in the cdR FoodLab Junior rapid test, which is equivalent to 10 µL of oil in 1000 µL hexane. The resulting UV-VIS spectra (absorption) can be seen from Figure 35.

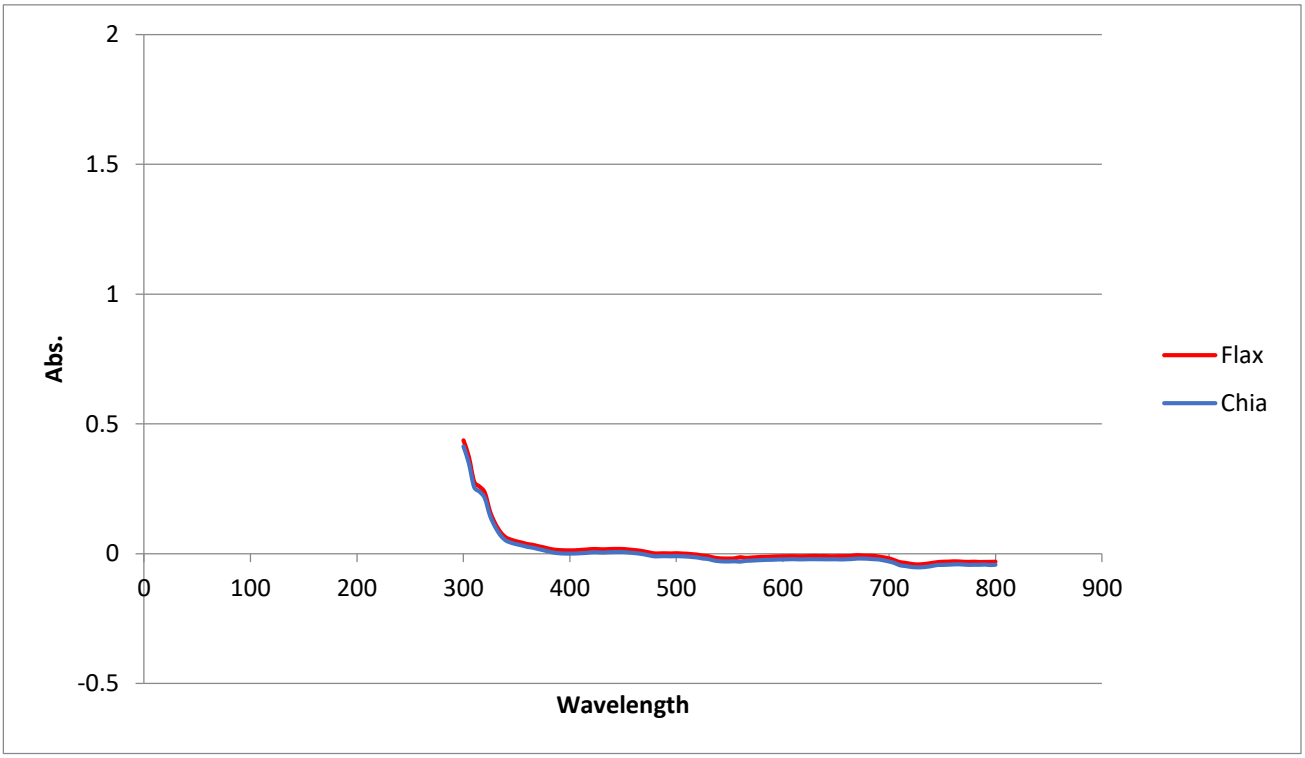


Figure 35: UV VIS spectra of chia and flaxseed oil in solution (1:1000) with hexane

The dilution was also measured at 505 nm and a UV VIS Spectrum was determined. Further on the determination of the peroxide value was done with the reagent kits of cdR at the FoodLab Junior and immediately after the measurements the reaction solution was transferred into micro cuvettes and measured at 505 nm. A UV VIS Spectrum of theses samples was determined as well, which can be seen from Figure 36 for chia seed oil and from Figure 37 for flaxseed oil. The results showed, that the genuine colour of the different oils has no influence on the measurements as in the final dilutions, the influence of the genuine colour of the oils did not show relevant absorption at 505 nm. With these experiments it is shown, that at present stage this rapid test is not suitable for the determination of fat classification numbers for the tested oils. The reasons of interference are not clear at present stage; an interference of secondary plant metabolites (e.g.) with high antioxidative properties might be one reason.

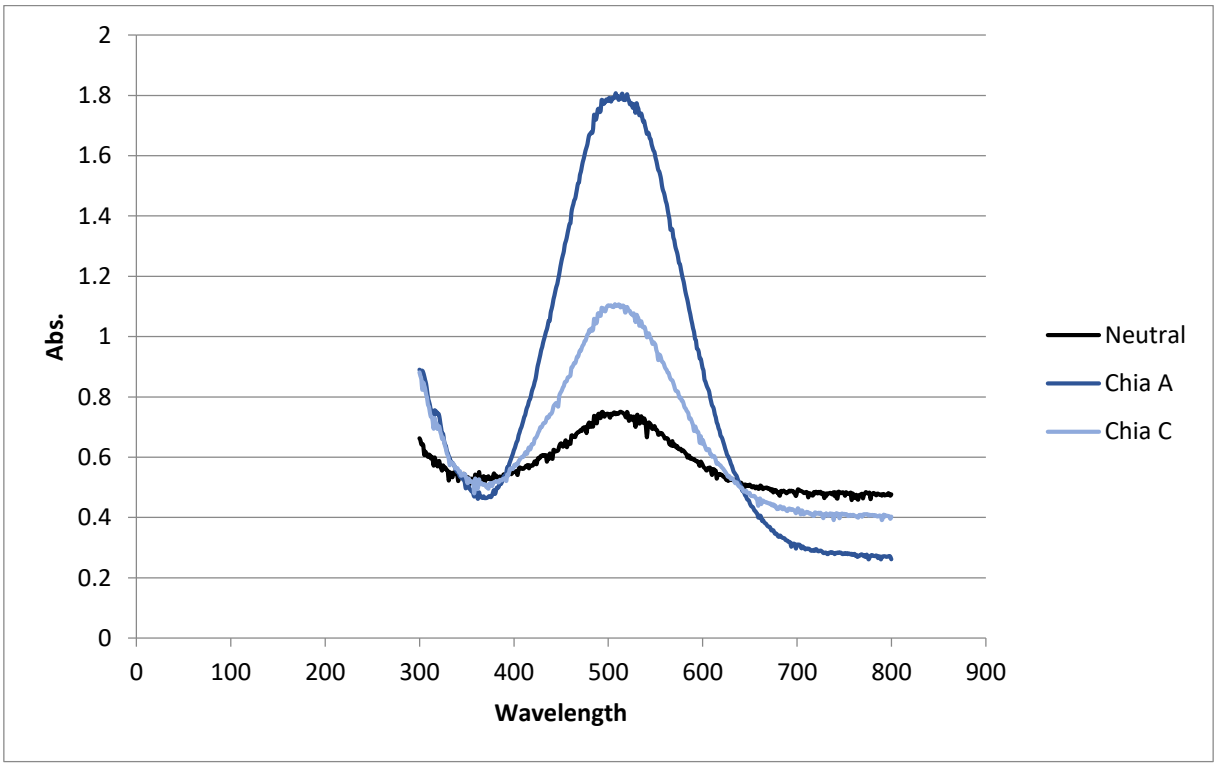


Figure 36: Comparison of the UV VIS spectra (absorption) of the cdR FoodLab Junior rapid test solutions for the determination of the peroxide value; chia seed oil A and C were used as well as the proposed blank value

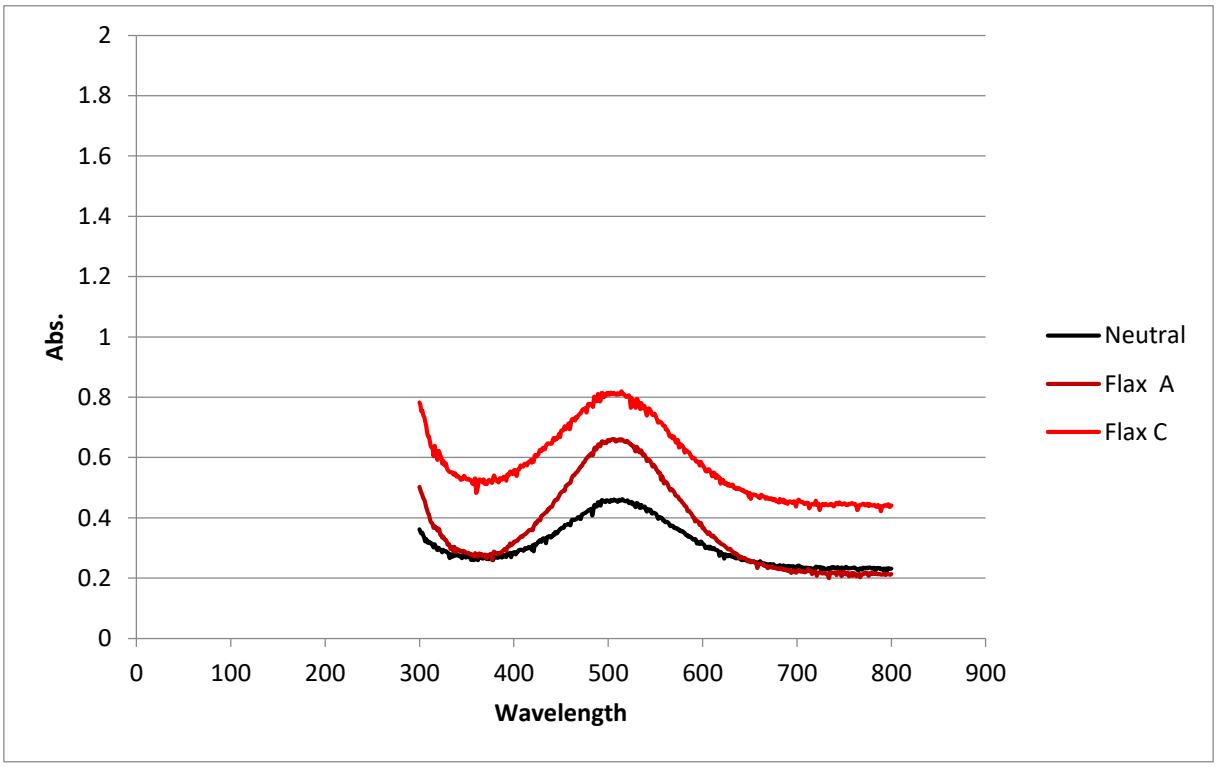


Figure 37: Comparison of the UV VIS spectra (absorption) of the cdR FoodLab Junior rapid test solutions for the determination of the peroxide value; flaxseed oil A and C were used as well as the proposed blank value

5.2. Investigation of the roasting, pressing and storage process of selected oils

5.2.1. Roasting and pressing of the tested oils

As the aim of this study was to show the changes in the different oil seeds and final pressed oils over the course of roasting, pressing and storage we tried to identify the volatile compounds found over different process steps in seeds as well as pressed oil. Additionally, we aimed to identify the compounds crucial for the formation of key flavour compounds of the investigated oils. The oils are stored in bigger tanks prior to filling to let the solid parts settle. One day after pressing the flaxseed oil is clear and can be filled. However, the chia seed oil is stored in a tank for approx. four weeks to let the solid parts settle to the ground and produce a clear oil, then the oil is filled into the final bottles. It takes approx. two weeks for the solid hemp parts to move to the ground and produce a clear oil that is then filled into bottles. The bottles are stored in a cool temperature storage unit until they are either sold or transported to a reseller. Also, to the measurements obtained from these samples an internal standard (2-pentanol) was added. As described above for the chia seed samples the concentrations and Odour Activity Values (OAV) for the samples were calculated in the same manner. The odour thresholds and the retention indices can be found in the according tables. Compounds with an OAV over 1 (or slightly beneath) were selected and can be seen in correlation with the temperature curve of the roasting process in the following figures. The detailed concentrations and OAVs can be found in the Appendix.

In the roasted **chia seeds** 1 acid, 7 alcohols, 1 aldehyde, 1 alkane, 8 esters, 1 furan derivative, 3 ketones, 4 terpenes and 2 other compounds were identified. The roasted chia seeds show an increasing concentration of D-limonene that peaks at the squeezing of the seeds. D-limonene, which is cyclic terpene, has a described flavour of fresh and sweet oranges [57]. Furthermore, alcohols like 1-propanol or 3-methyl-1-butanol as well as the acids like heptanoic or acetic acid peak in the squeezed seeds. 3-methyl-1-butanol has been reported in olive oil to add to a fusty/muddy sediment defect, if found in high concentrations [59]. The determination of acetic acid was expected, as this acid is formed while processing the seeds [60]. Propanal is believed to oxidize to propanoic acid and exceeds as a product of oxidation. Also, hexanal can be formed as the oxidation continues [61]. Hexanal has been found to be one of the most often named oxidation markers for evaluation of off-flavour in different kinds of food. Nevertheless, it is only connected to the development and progress of rancidity and not directly to the off-flavour. Biplot A in

Figure 39 shows the volatiles determined in the roasting steps of the chia seeds. The samples that are found close to each other impose a similarity in their aroma profiles; it can be seen that those compounds change over the whole process. In the roasting process, the volatile compounds found in the raw material and the squeezed seeds are correlating (see Quadrants I and IV). In the raw material, mainly volatile esters are found, compared to the further steps of the roasting process (see Quadrant IV). In the squeezed seeds, the first fat oxidation products are found (see Quadrant I). The compounds change over the whole roasting process. But as the roasting process goes further on aldehydes and alcohols can be found mainly. Compared to the relative concentrations (given in the Appendix) the concentrations of the volatile compounds do not increase nor decrease after the roasting time of 10 respectively 15 minutes (see Quadrant III).

In the pressed and filled **chia seed oil** 1 acid, 3 alcohols, 1 aldehyde, 2 ketone, 2 terpene and 1 other compounds were identified. However, looking at the pressed oil the chia seed oil shows a high concentration in acetic acid.

The determination of acetic acid was expected, as the acetic acid is formed during processing seeds, no matter which kind of seeds are processed [60]. Alcohols like 2-methyl-1-butanol and 3-methyl-1-butanol have been reported in other kinds of vegetable oils, for example, in flax seed oil by Ivanova-Petropulos *et al.* [60] in 2015 as well as in cold pressed native rape seed oil by Matthaeus *et al.* in 2002

[63]. The odour of benzaldehyde has been reported to be bitter almond like and has been found by different authors like Ivanova-Petropulos *et al.* [60] in 2015 in flax seed oil as well as Taticchi *et al.* [64] in 2013 in olive oil. Benzaldehyde is also known as one of the Strecker aldehydes, which are most likely derived from the amino acid precursor's valine, isoleucine, leucine and alanine. Intense thermal treatment is required to generate these aldehydes as they were only reported in oil samples with a roasting time of over 20-30 minutes [65]. Additionally, the obtained data for all oils was evaluated statistically. Therefore, biplot analysis were run to give a graphical deception. As seen in biplot B from Figure 39 the freshly pressed oil differs significantly from the other three measurement points. Mostly fat oxidations products are responsible for these differences. As these compounds are already found in the samples one day after pressing and over the storage period in the tank, those fat oxidation products could form while the oil is settling in the tanks (see Quadrant I). Interestingly, the samples taken from the storage of the oil in bigger tanks and one day after the filling of the oil can be correlated with Strecker aldehydes mainly (see Quadrant IV). Overall no significant correlation between the four samples obtained over the storage process can be seen.

The comparison of the OAV value of selected volatiles over the whole roasting and storage process after pressing is shown in Figure 38. If the according odour threshold value for a certain compound was available in oil the OAV was calculated. By far the highest OAV was calculated for acetic acid and therefore has a great impact on the overall flavour. Compared to 1-hexanol, having a green, tallow, fat and flower like smell, with a relatively low odour threshold (0.12 mg/kg) is more likely to contribute to the overall aroma. Benzaldeyhde having an even more low odour threshold (0.06 mg/kg) gives an almond-like and burnt smell and according to the raised OAV contributes to the overall odour impression. 3-methyl-1-butanol reported to have a whiskey, malt and burnt odour is a precursor for the according aldehydes and their oxidation is likely to occur under the roasting conditions [66].

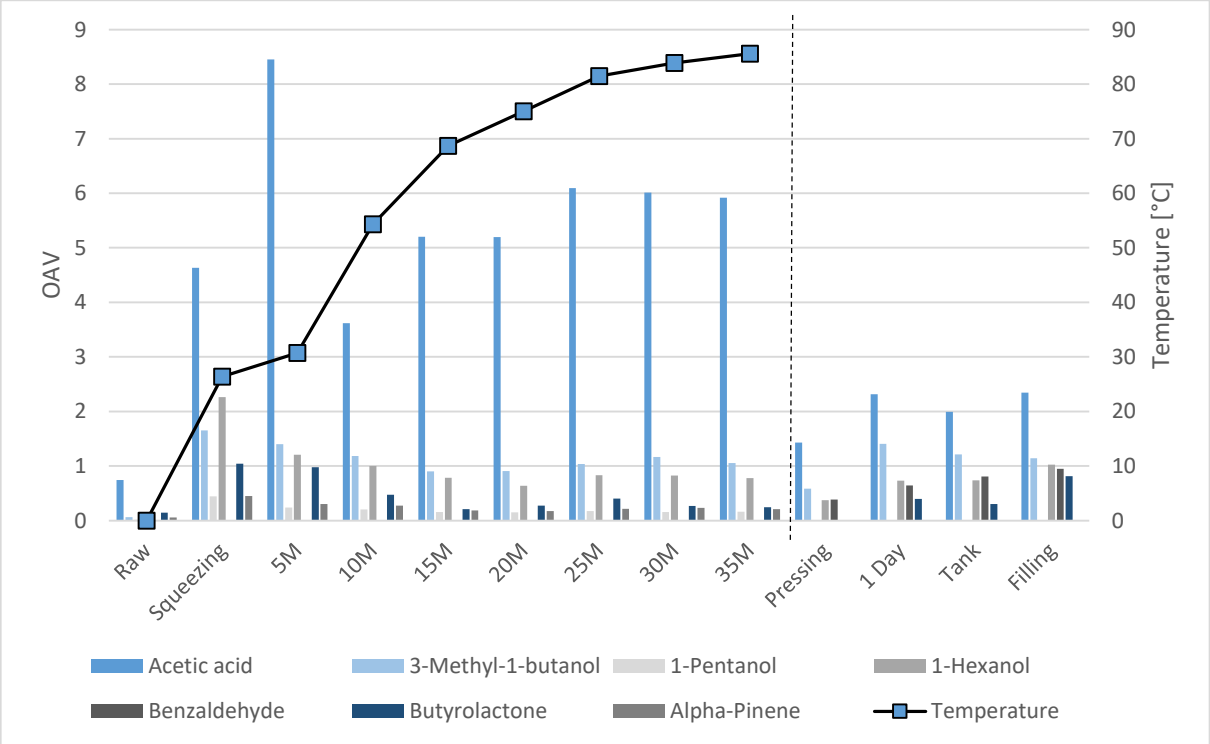
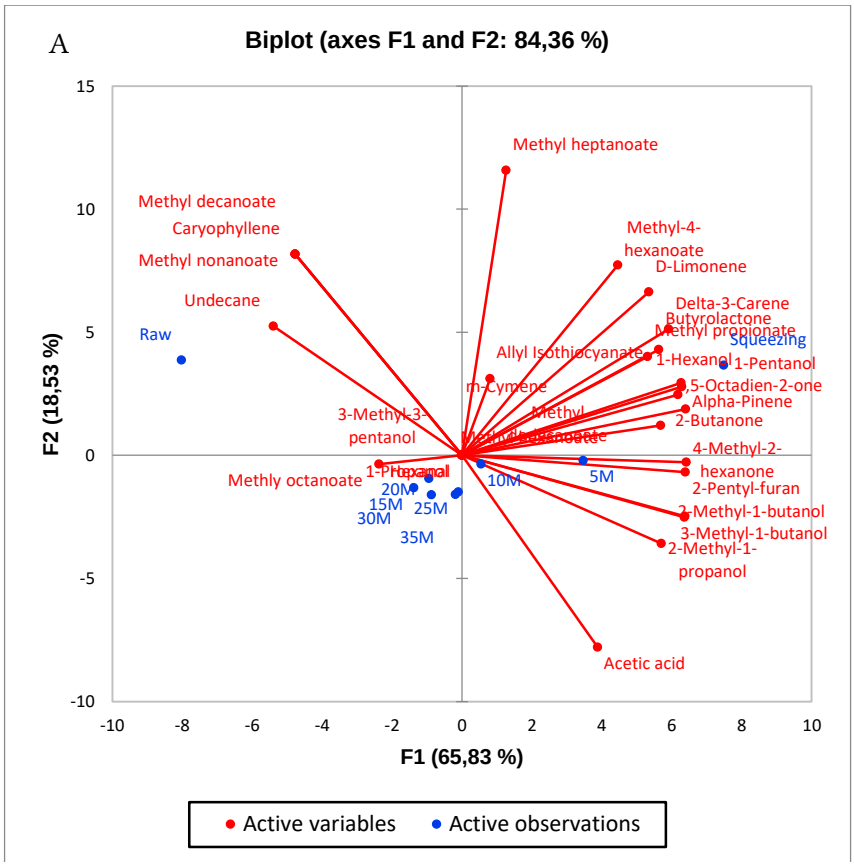


Figure 38: Comparison of the determined OAV values of selected compounds in the roasting, pressing and storage and the increasing surface temperature while roasting of chia seed oil



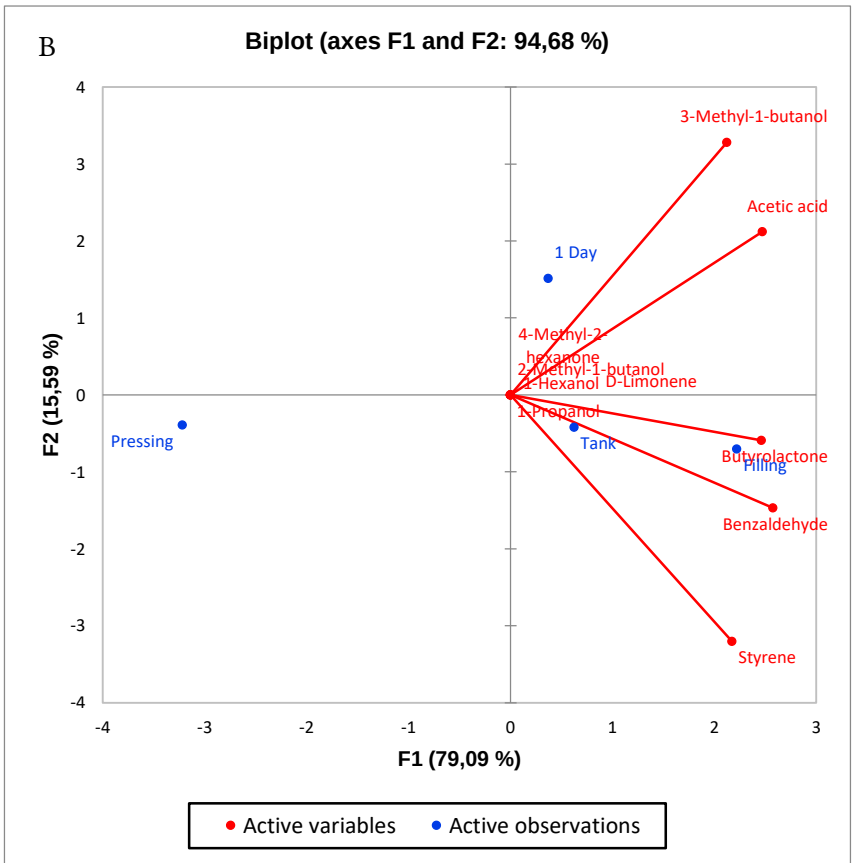


Figure 39: Biplot analysis of the roasting process of chia seeds with the samples indicated in blue and the determined volatiles using SPME GC-MS indicated in red, calculated values of the concentration relative to the internal standard in mg/kg of measured samples were used (n=4) – (A): roasting process of chia seed oil, (B): storage process of chia seed oil

Table 16: Volatile compounds identified in the roasting and pressing process of chia seeds (samples were collected on the 30.08.2017 and measured on the 13.09.2017) calculated RI value in comparison to the RI Lit values found in literature (n=4) and the odour thresholds found in oil are given

Compound	S/N	rt time [min]	RI (HP5)	RI Lit	Odour threshold [mg/kg]	Odour description	Identified in
Acids							
Acetic acid		7.16	684	600[1]	0.75	sour	A, B, C, D, E, F, G, H, I, J, K, L, M
Alcohols							
1-Propanol	n.q.	4.74	594	536[1]		alcohol, pungent	B, C, D, E, F, G, H, I
2-Methyl-1-propanol		6.13	643	636[2]	1	etheral, winey (3)	B, C, D, E, F, G, H, I
3-Methyl-1-butanol		8.64	743	736[1]	0.1	whiskey, malt, burnt	A, B, C, D, E, F, G, H, I, J, K, L, M
2-Methyl-1-butanol	n.q. (in Oil)	8.72	746	739[1]	0.48	malt	A, B, C, D, E, F, G, H, I, J, K, L, M
3-Methyl-3-pentanol	n.q.	9.05	759[t]			fruity, green, leafy	A, B, C, D, E, F, G, H, I
1-Pentanol		9.40	773	775[2]	0.47	balsamic	A, B, C, D, E, F, G, H, I
1-Hexanol	n.q. (in Oil)	11.77	871	851[1]	0.4	resin, flower, green	A, B, C, D, E, F, G, H, I, J, K, L, M
Aldehydes							
Hexanal	n.q.	10.13	802	801[1]	0.12	grass, tallow, fat	A, B, C, E, G, H, I
Benzaldehyde		13.84	962	960[1]	0.06	almond, burnt suger	J, K, L, M
Alkanes							
Undecane		16.73	1100	1100[1]	5.75	alkane	A, C, D, E, F, G, H, I
Esters							
Methyl propionate		5.91	634	621[2]		fresh, rummy, fruity, strawberry, apple[3]	B, C, D, E, F, G, I
Methyl hexanoate	n.q.	13.00	924	931[2]		fruity, pineapple, ether[3]	A, B, C, D, E, F, G, H, I
Methyl-4-hexanoate		13.14	931[t]				B
Methyl heptanoate		15.15	1023	1021[2]		sweet, fruity, green, waxy, apple[3]	A, B
Methly octanoate		17.23	1126	1126[2]		waxy, green, sweet, orange, aldehydic, vegetable, herbal[3]	A, D, E, F, H, I
Methyl nonanoate		19.06	1223	1227[2]		sweet, fruity, pear, waxy, winey, tropical[3]	A
Methyl decanoate		20.84	1324	1373[1]		rancid fat	A

Compound	S/N	rt time [min]	RI (HP5)	RI Lit	Odour threshold [mg/kg]	Odour description	Identified in
Methyl dodecanoate	n.q.	24.09	1523	1526[2]		waxy, soapy, creamy, coconut, mushroom	A
Furan derivatives							
2-Pentyl-furan		14.48	991	993[1]	8	green bean, butter	A, B, C, D, E, F, G, H, I
Ketones							
4-Methyl-2-hexanone	n.q. (in Oil)	11.21	847	792[1]		ether	A, B, C, D, E, F, G, H, I, J, K, L, M
Butyrolactone		12.71	911	908[2]	0.3	creamy, oily, fatty[3]	A, B, C, D, E, F, G, H, I, K, L, M
***(E,E)*-3,5-Octadien-2-one**		16.11	1070	1072[2]		fatty, fruity, hay, green, herbal [3]	A, B, C, D, E, F, G, H, I
Other compounds							
Allyl Isothiocyanate		12.00	881	887[1]		sulfur, pungent, garlic	A, B, C, D, E, F, G, H, I
Caryophyllene		22.63	1430	1418[2]		sweet, wood, spicy, dove, dry[3]	A
Styrene		12.26	891	893[1]	7.65	balsamic, gasoline	J, K, L, M
Terpenes							
α-Pinene		13.23	934	939[1]	0.274	pine, turpentine	A, B, C, D, E, F, G, H, I
δ-3-Carene		14.89	1010	1009[1]		lemon, resin	A, B, C, D, E, F, G, H, I
m-Cymene	n.q., n.d. (in Oil	15.22	1026	1027[1]	2.51*	solvent, citurs, gasoline	A, B, C, D, E, F, G, H, I, J, K, L, M
D-Limonene	n.q. (in Oil)	15.32	1031	1030[1]	14.7	citurs, mint	A, B, C, D, E, F, G, H, I, J, K, L, M

A = Raw material, B = squeezed seeds, C = roasting 5 min., D = roasting 10 min., E = roasting 15 min., F = roasting 20 min., G = roasting 25 min., H = roasting 30 min., I = roasting 35 min., J = freshly pressed oil, K = oil one day after pressing, L = oil after storage in a tank, M = oil after filling the final bottle

S/N – Signal/Noise Ratio, n.d. - not detectable (S/N < 3), n.q. – not quanitifable (S/N < 10)

RI – Retention Index

RI (HP5) – RI as detemerined in the measurments

RI Lit – referencened RI obtained from databases

[1] – RI obtained from http://www.flavornet.org

[2] – RI obtained from http://webbook.nist.gov/

[3]– Odour description obtained from http://www.thegoodscentscompany.com/

[t] - Tentatively identified, tentative identification is based on the mass spectra and RIs obtained from RI databases

* - OAV determined experimentally

Odour tresholds were collected from "Odour tresholds: compilations of odour threshold values in air, water and other media" – L.J. van Gemert, using the matrix oil/vegetable oil

In the roasted **flaxseeds** 9 alcohols, 2 aldehydes, 4 esters, 1 furan derivatives, 3 ketones, 4 terpenes and 2 other compounds were identified. In the roasting process of the flaxseeds a decreasing concentration in α-pinene was observed. The odour of α-pinene is described as sharp, pine, turpentine and grassy [67]. Ivanova-Petropulos *et al.* [60] reported in 2015 α-pinene in flax seed oil and that high concentrations of this compound can lead to negative effect on the overall flavour characteristic. When looking at the relative concentration of 1-pentanol, a slight peak at the squeezing of the seeds is seen, but the concentration is then quite equal over the roasting process itself. In the obtained flaxseed samples, the elevation of 1-pentanol could be observed, as well as a higher concentration of 1-hexanol and 2-methyl-1-propanol. 1-Hexanol has been reported to have an herbaceous, woody and green odour and is, as it is one of the main volatile compounds, contributing to the characteristic odour of flaxseed oil [17]. Alcohols like 2-methyl-1-butanol and 3-methyl-1-butanol have been reported, for example, in flaxseed oil by Ivanova-Petropulos *et al.* [60] in 2015 as well as in cold pressed native rape seed oil by Matthaeus *et al.* in 2002 [63].
However, in the pressed and filled **flaxseed oil** 6 alcohols, 1 terpene and 2 other compound were identified Looking at the freshly pressed flaxseed oil and the oil after filling, also 1-hexanol and 2-methyl-1-propanol show a higher concentration. Furthermore, the concentration of 1-pentanol is also elevated in the pressed and filled oil. The concentration of hexanal in the freshly pressed oil is twice as much as in the filled oil. Alcohols, like 2-methyl-1-butanol and 3-mehtyl-1-butanol, have been described above. ThFrom the samples of the flaxseeds and flaxseed oil biplot analysis were done as seen in
Figure 43. Also, here the raw material shows overall the biggest difference in the volatile composition compared to the squeezed and roasted seeds (see Quadrant IV). Over the process of roasting the composition of the compounds is not changing significantly. It could be observed, that the two oil samples are different from the roasted seeds, but the aroma profiles for the freshly pressed and filled oil are correlating (see Quadrant III). It could be possible that the volatile compounds found in the roasted seeds but not found in the oil anymore could got lost through the pressing cake. Looking also at the relative concentrations (given in the Appendix) the concentrations of the volatile compounds do not further increase nor decrease after the roasting time of 10 respectively 15 minutes (see Quadrant III).
OAV obtained from the determined volatiles compared to the increasing surface temperature over the roasting, pressing and storage process has been compared is shown in Figure 40. 2-methyl-1-propanol was found in the filled oil and therefore contributes, especially in the filled oil, to the overall aroma. 1-Hexanol gives a flower, green and resin-like odour and has been found to contribute to the overall flavour, with a rather low odour threshold (0.4 mg/kg), the highest concentration could be found in the freshly pressed oil. α-Pinene is described with a pine, turpentine like odour and has a relatively low odour threshold (0.274 mg/kg), it shows increasing values over the roasting process.

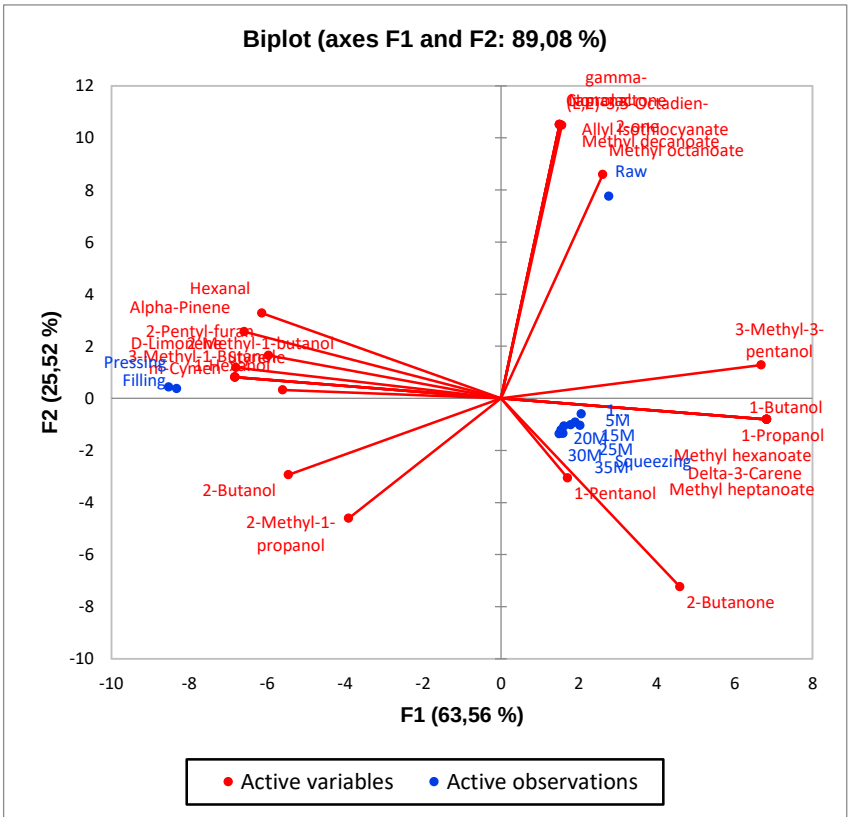


Figure 40: Biplot analysis of the roasting, pressing and filling process of flaxseeds and flaxseed oil with the samples indicated in blue and the determined volatiles using SPME GC-MS indicated in red, calculated values of the concentration relative to the internal standard of the concentration in mg/kg of measured samples were used (n=4)

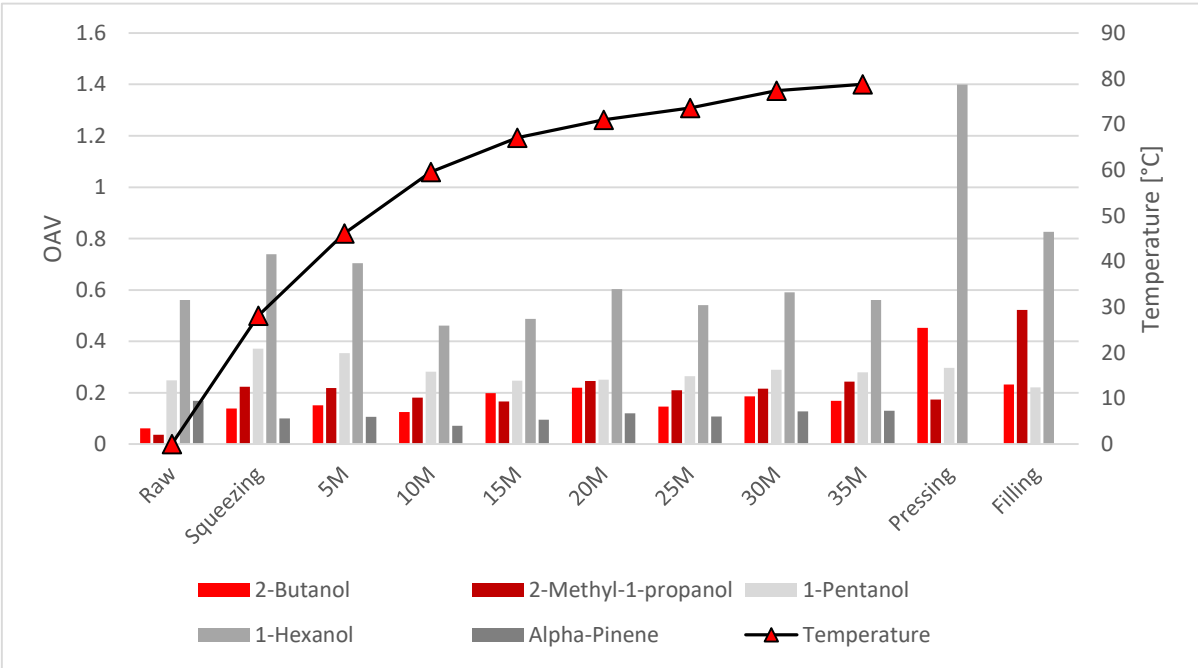


Figure 41: Comparison of the determined OAV values of selected compounds in the roasting, pressing and storage and the increasing surface temperature while roasting of flaxseed oil

Table 17: Volatile compounds identified in the roasting and pressing process of flaxseeds (samples were collected on the 12.09.2017 and measured on the 14.09.2017) calculated RI value in comparison to the RI Lit values found in literature (n=4) and the odour thresholds found in oil are given

Compound	S/N	rt time [min]	RI (HP5)	RI Lit	Odour threshold [mg/kg]	Odour description	Identified in
Alcohols							
1-Propanol	n.q.	4.78	595	536[1]		alcohol, pungent	A, B, C, D, E, F, G, H, I
2-Butanol		5.58	621	612[2]	0.5	etheral[3]	A, B, C, D, E, F, G, H, I, J, K
2-Methyl-1-propanol		6.16	644	636[2]	1	fermented[3]	A, B, C, D, E, F, G, H, I, J, K
1-Butanol	n.q.	6.98	677	675[1]	0.038	medicine, fruit	B, C, D, E, F, G, H, I
3-Methyl-1-Butanol	n.q.	8.65	743	736[1]	0.1	whiskey, malt, burnt	A, B, C, D, E, F, G, H, I, J, K
2-Methyl-1-butanol	n.q.	8.73	746	739[1]	0.48	malt	A, B, C, D, E, F, G, H, I, J, K
3-Methyl-3-pentanol		9.06	759[t]			fruity, green, leafy[3]	A, B, C, D, E, F, G, H, I
1-Pentanol		9.40	773	775[2]	0.47	balsamic	A, B, C, D, E, F, G, H, I, J, K
1-Hexanol		11.75	870	869[2]	0.4	resin, flower, green	A, B, C, D, E, F, G, H, I, J, K
Aldehydes							
Hexanal	n.q. (in Oil)	10.13	802	801[1]	0.12	grass, tallow, fat	A, B, C, D
Nonanal		16.80	1104	1104[1]	1	fat, citrus, green	A, B
Esters							
Methyl hexanoate	n.q.	13.00	924	931[2]		fruity, pineapple, etheral[3]	A, B, C, D, E, F, G, H, I
Methyl heptanoate	n.q.	15.13	1022	1021[2]		sweet, fruity, green, waxy, apple[3]	A, B, C, D, G, H, I
Methyl octanoate		17.14	1121	1279[1]		sweat, cheese	A, B, C, D
Methyl decanoate		20.62	1312	1373[1]		rancid fat	A
Furan derivatives							
2-Pentyl-furan		14.46	990	993[1]	8	green bean, butter	A, B, C, D, E, F, G, H, I
Ketones							
2-Butanone		5.15	604	602[2]	40	etheral, fruity, campherous[3]	B, C, D, E, F, G, H, I
γ-Caprolactone	n.q.	15.79	1054	1056[2]		creamy, oily, fatty, caramellic[3]	A
***(E,E)*-3,5-Octadien-2-one**		16.10	1069	1072[2]		fatty, fruity, hay, green, herbal[3]	A
Other compounds							
Styrene	n.q.	12.50	902	893[1]	7.65	balsamic, gasoline	A, B, C, D, E, F, G, H, I, J, K
Allyl Isothiocyanate		12.02	881	887[1]		sulfur, pungent, garlic	A

Compound	S/N	rt time [min]	RI (HP5)	RI Lit	Odour threshold [mg/kg]	Odour description	Identified in
Terpenes							
α-Pinene	n.q. (in Oil)	13.23	934	939[1]	0.274	pine, turpentine	A, B, C, D, E, F, G, H, I, J, K
δ-3-Carene	n.q.	14.88	1010	1009[1]		lemon, resin	A, B, C, D, E, F, G, H, I
m-Cymene	n.q.	15.22	1026	1027[1]	2.51[*]	solvent, gasolien, citrus	A, B, C, D, E, F, G, J, K
D-Limonene	n.q. (in Oil)	15.32	1031	1030[1]	14.7	citurs, mint	A, B, C, D, E, F, G, H, I, J, K

A = Raw material, B = squeezed seeds, C = roasting 5 min., D = roasting 10 min., E = roasting 15 min., F = roasting 20 min., G = roasting 25 min., H = roasting 30 min., I = roasting 35 min., J = freshly pressed oil, K = oil one day after pressing/filling

S/N – Signal/Noise Ratio, n.d. - not detectable (S/N < 3), n.q. – not quanitifable (S/N < 10)

RI – Retention Index

RI (HP5) – RI as determined in the measurements

RI Lit – referenced RI obtained from databases

[1] – RI obtained from http://www.flavornet.org

[2] – RI obtained from http://webbook.nist.gov/

[3] – Odour description obtained from http://www.thegoodscentscompany.com/

[t] - Tentatively identified, tentative identification is based on the mass spectra and RIs obtained from RI databases

[*] - OAV determined experimentally

Odour thresholds were collected from "Odour thresholds: compilations of odour threshold values in air, water and other media" – L.J. van Gemert, using the matrix oil/vegetable oil

Looking at the roasted **hempseeds** 1 acid, 7 alcohols, 9 aldehydes, 5 esters, 6 ketones, 11 terpenes, 7 pyrazines and 3 other compounds were identified. In the raw and squeezed hempseeds higher concentrations of 3-methyl-1-butanol, 1-pentanol, hexanoic acid methyl ester and β -myrcene could be detected. The concentration for 3-methyl-1-butanol was decreasing over the time of roasting. Also, the concentration of 1-hexanol decreased steady over the time of roasting, as well as the concentration of 2-heptanol, hexanoic acid methyl ester, β -myrcene and 3-ethyl-2,5-dimethyl-pyrazine. The odour of 2-heptanol has been described as fruity, sour and green, in roasted palm kernel oil this compound was found to be aroma-active in contrast to the unroasted oil [65]. The concentration of hexanal was peaking after 15 minutes of roasting and decreased slightly until the end of roasting. The concentration of benzaldehyde increased over the time course of roasting. Also, for the hempseeds a statistical biplot analysis was done, as seen in
Figure 43, biplot A. Here the raw material and squeezed seeds are quite similar in their volatile composition (see Quadrant IV). Over the roasting process more changes can be seen, as pyrazines start to form (see Quadrants IV, I and II).
In the pressed and stored **hempseed oil** 2 alcohols, 6 aldehydes, 1 alkane, 2 alkenes, 1 furan derivative, 2 ketones, 7 terpenes, 6 pyrazines and 1 other compounds were determined. Taking a closer look at the pressing and storage process of the hempseed oil, it can be said that the concentration of compounds like 3-methyl-butanal and 2-mehtyl-butanal or hexanal or benzaldehyde peaked at the measurement point of the storage in the tank. These compounds derive from the degradation of amino acids and are often termed as Strecker aldehydes. Hexanal has been found to be one of the most often named oxidation markers for evaluation of off-flavour in different kinds of food. Nevertheless, it is only connected to the development and progress of rancidity and not directly to the off-flavour. Additionally, other aldehydes, such as, pentanal or nonanal have been reported to be correlated with rancidity [62]. The odour of benzaldehyde has been reported to be bitter almond like and has been found by different authors like Ivanova-Petropulos *et al.* [60] in 2015 in flax seed oil as well as Taticchi *et al.* in 2013 in olive oil [64]. Benzaldehyde, 2-methyl-butanal or 3-methyl-butanal are also known as Strecker aldehydes, which were most likely derived from the amino acid precursor's valine, isoleucine, leucine and alanine. Intense thermal treatment is required to generate these aldehydes as they were only reported in oil samples with a roasting time of over 20-30 minutes [65]. Furthermore, also flavour-forming compounds like methyl-pyrazine or α-pinene peaked in the sample after the storage in a tank. Other pyrazines like 3-ethyl-2,5-dimethyl-pyrazine peaked one day after the pressing and decreased then quite drastic until the filling of the oil. The appearance of different pyrazines, such as methyl-pyrazine has been reported by Siegmund and Murkovic [66] in 2004 in the roasting process of pumpkin seed oil. In this study the formation of different pyrazines has been observed at temperatures above 100°C and a minimum reaction time of 50 minutes. These compounds have also been found to be correlation with the sensory attributes of roasty and nutty [66]. Ivanova-Petropulos *et al.* [60] reported in 2015 α-pinene in different variants of flax seed oil and that high concentrations of this compound can lead to negative effect on the overall flavour characteristic [64]. In rather low concentrations the terpene compound has a relatively high odour intensity and can lead to a turpentine, pine-like or sharp odour [67]. In biplot B from
Figure 43, the freshly pressed oil is different in composition than the roasted seeds and other (or less) volatiles compounds are found (see Quadrant I). It is possible that these compounds can be found in the pressing cake. The fat oxidation products are formed over the storage period in bigger tanks while the oil is settling (see Quadrant IV).
This shows that the time of 40 minutes for the roasting of the hempseeds, but also the high temperatures as mentioned above, are needed to form pyrazines, that give the final product the typical odour and taste.

The calculated OAV values compared to the increasing surface temperature over the roasting, pressing and storage process is shown in Figure 42. As shown in Figure 42 the forming of pyrazines increases with the increased roasting temperature as well as over the course of time.
The grass, tallow and fat-smelling hexanal peaks in the tank storage of the oil with an OAV over 9. Trimethylpyrazine with a rather low odour threshold (0.027 mg/kg) contributing to the overall odour impression. 2,5-Dimethylpyrazine a high odour threshold (2.6 mg/kg) and are therefore less likely to contribute to the aroma. The smell of pyrazines is mostly described as a roasted, nutty, potato like smell. The increase of the pyrazines over the time of roasting and their increasing contribution to the overall aroma can be seen.

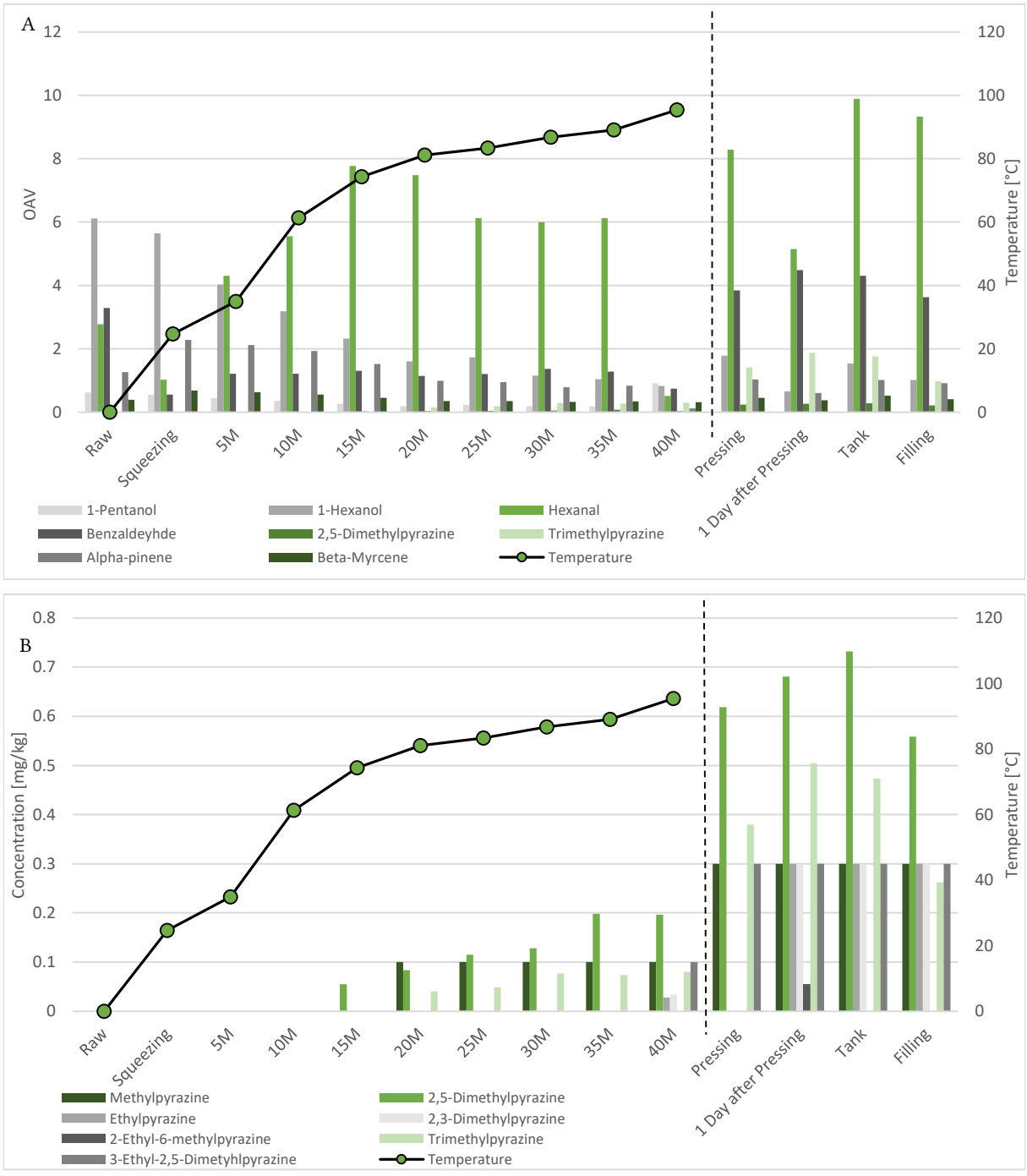


Figure 42: Comparison of OAV/calculated concentrations and the roasting temperature in the roasting, storage and pressing process of hempseed oil production (A) : Comparison of the determined OAV values of selected compounds, (B) Comparison of the calculated concentrations of all found pyrazines in hempseeds and oil

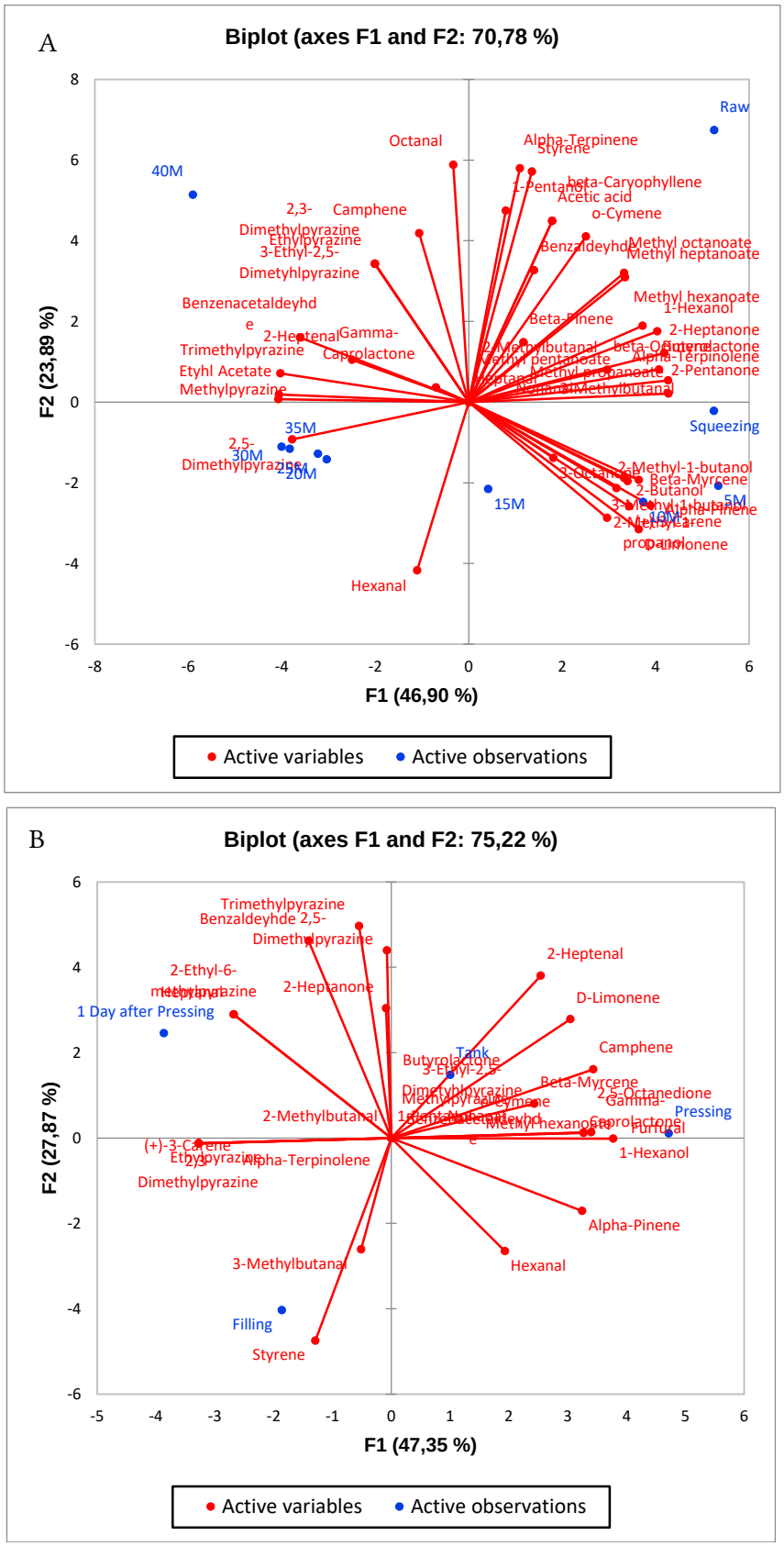


Figure 43: Biplot analysis of the roasting process of hempseed oil with the samples indicated in blue and the determined volatiles using SPME GC-MS indicated in red, calculated values of the concentration relative to the internal standard of the concentration in mg/kg of measured samples were used (n=4) - (A): roasting of hempseed oil, (B): storage of hemp seed oil)

Table 18: Volatile compounds identified in the roasting and pressing process of hempseeds (samples were collected on the 27.09.2017 and measured on the 09.10.2017) calculated RI value in comparison to the RI Lit values found in literature (n=4) and the odour thresholds found in oil are given

Compound	S/N	rt time [min]	RI (HP5)	RI Lit	Odour threshold [mg/kg]	Odour description	Identified in
Acids							
Acetic acid	n.q.	6.78	669	600[1]	0.75	sour	A
Alcohols							
1-Propanol	n.d.	4.70	593	536[1]		alcohol, pungent	A, B, C, D, E
2-Butanol		5.53	619	612[2]	0.5	sweet, apricot[3]	A, B, C, D, E, F, I
2-Methyl-1-propanol		6.11	642	636[2]	1	etheral, winey[3]	B, C, D, E
3-Methyl-1-butanol		8.64	743	736[1]	0.1	whiskey, malt, burnt	A, B, C, D, E, G
2-Methyl-1-butanol		8.72	746	739[1]	0.48	malt	A, B, C, D, E, G
1-Pentanol	n.q. (in Oil)	9.40	773	775[2]	0.47	balsamic	A, B, C, D, E, F, G, H, I, J, K, L, M,N
1-Hexanol		11.78	871	869[2]	0.4	etheral, fusel oil, fruity, alcoholic, sweet, green[3]	A, B, C, D, E, F, G, H, I, J, K, L, M,N
Aldehydes							
3-Methylbutanal	n.q.	6.68	665	650[1]	0.0054	malt	F, G, J, M, N
2-Methylbutanal	n.q.	6.77	669	641[1]	0.0052	cocoa, almond	F, G, J, K, L, M,N
Hexanal		10.13	802	801[1]	0.12	grass, tallow, fat	A, B, C, D, E, F, G, H, I, J, K, L, M,N
Heptanal	n.q.	12.52	903	903[1]	0.25	fat, citrus, rancid	L
2-Heptenal		13.74	958	957[1]	1.5	soap, fat, almond	A, E, F, G, H, I, J, K, L, M,N
Benzaldeyhde		13.88	964	960[1]	0.06	almond, burnt sugar	A, B, C, D, E, F, G, H, I, J, K, L, M,N
Octanal		14.84	1008	1006[1]	0.32	fat, lemon, soap, green	A
Benzenacetaldeyhde	n.q. (J & in Oil)	15.63	1046	1039[2]	0.04*	green, sweet, honey, floral[3]	D, E, F, G, H, I, J
Nonanal	n.q.	16.83	1105	1104[1]	1	fat, citrus, green	A, F, G, H, I, J, K, L, M,N
Esters							
Methyl propanoate	n.q.	5.97	636	621[2]		fresh, rummy, fruity, strawberry, apple[3]	A, B
Methyl pentanoate	n.q.	10.71	826	823[2]		sweet, green, fruity, apple, pineapple, nutty	A, B, C

Compound	S/N	rt time [min]	RI (HP5)	RI Lit	Odour threshold [mg/kg]	Odour description	Identified in
Methyl hexanoate	n.q. (in Oil)	13.02	925	931[2]		fruity, pineapple, etheral[3]	A, B, C, D, E, F, G, H, I, J, K, L, M,N
Methyl heptanoate		15.18	1024	1021[2]		sweet, fruity, green, orris, waxy, floral, berry[3]	A, B, C
Methyl octanoate		17.17	1123	1279[1]		waxy, green, sweet, orange, aldehydic, vegetable, herbal[3]	A, B, C
Ketones							
2-Pentanone	n.q.	7.23	687	711[1]		ether, fruit	A, B, C, D, E
2-Heptanone	n.q. (A,B)	12.22	890	895[1]	0.3	soap	A, B, C, D, E, F, G, H, I, J, K, L, M,N
Butyrolactone	n.q.	12.72	911	908[2]		creamy, oily, fatty, caramellic[3]	A, B, C, D, K, L, M,N
2,5-Octanedione	n.q. (in Oil)	14.34	985	988[1]		earth, must	K
3-Octanone	n.q.	14.35	985	984[2]		herb, butter, resin	C
γ-Caprolactone	n.q. (in Oil)	15.77	1053	1056[2]		creamy, oily, fatty, caramellic[3]	B, C, D, K
Other compounds							
Styrene	n.q. (in Oil)	12.36	896	893[1]	7.65	balsamic, gasoline	A, B, N
Etyhl Acetate	n.q. (J)	5.60	622	618[2]	0.94	etheral, fruity, sweet, weedy, green[3]	E, F, G, H, I, J
Fufural		10.88	833	829[1]		bread, almond, sweet	K, L, M,N
Pyrazines							
Methylpyrazine	n.q.	10.71	826	828[1]	27	popcorn	F, G, H, I, J, K, L, M,N
2,5-Dimethylpyrazine		12.74	912	913[1]	2.6	roasted nut, cocoa, roasted beef	E, F, G, H, I, J, K, L, M,N
Ethylpyrazine	n.q. (in Oil)	12.76	913	915[2]	17	nutty, musty, fermented, coffee, roasted, cocoa, meaty[3]	J, L, M, N
2,3-Dimethylpyrazine	n.q. (in Oil)	12.82	916	919[2]		musty, nut skin, cocoa, powdery, roasted, potato, coffee	J, L, M, N
2-Ethyl-6-methylpyrazine		14.62	997	997[2]		roasted potato[3]	L
Trimethylpyrazine		14.73	1003	1000[1]	0.27	roast, potato, must	F, G, H, I, J, K, L, M,N
3-Ethyl-2,5-Dimetyhlpyrazine	n.d. (I), n.q. (J & in Oil)	16.26	1077	1082[1]	0.024	potato, roast	I, J, K, L, M, N

Compound	S/N	rt time [min]	RI (HP5)	RI Lit	Odour threshold [mg/kg]	Odour description	Identified in
Terpenes							
α-Pinene		13.26	936	939[1]	0.274	pine, turpentine, camphor	A, B, C, D, E, F, G, H, I, J, K, L, M,N
Camphene	n.q. (in Oil)	13.61	952	953[1]		camphor	A, B, C, D, E, F, G, H, I, J, K, M
β-Pinene		14.24	980	981[1]	430	pine, resin, turpentine	A, B, C, D, E, F, G, H, I, J
β-Myrcene		14.46	990	992[1]	0.79[*]	balsamic, must, spice	A, B, C, D, E, F, G, H, I, J, K, L, M,N
δ-3-Carene	n.q. (in Oil)	14.90	1011	1009[1]		lemon, resin	A, B, C, D, E, F, G, H, I, J, L, M, N
α-Terpinene		15.07	1019	1012[1]		lemon	A, B, C, D
m-Cymene	n.q. (in Oil)	15.24	1027	1027[1]	2.51[*]	solvent, gasoline, citrus, eucalyptus, herbal, camphoreous	A, B, C, D, E, F, G, H, I, J, K, L, M,N
D-Limonene		15.34	1032	1033[1]	14.7	lemon, orange	A, B, C, D, E, F, G, H, I, J, K, L, M,N
β-Ocimene		15.73	1051	1038[1]		sweet, herb	A, B, C, D, E
α-Terpinolene	n.q. (in Oil)	16.49	1088	1090[1]		sweet, fresh, pine, citurs, woody, lemon peel[3]	A, B, C, D, E, F, G, H, I, J, L, M, N
β-Caryophyllene		22.64	1430	1467[1]		sweet, wood, spicy, dove, dry[3]	A

A = Raw material, B = squeezed seeds, C = roasting 5 min., D = roasting 10 min., E = roasting 15 min., F = roasting 20 min., G = roasting 25 min., H = roasting 30 min., I = roasting 35 min., J = roasting 40 min., K = freshly pressed oil, L = oil one day after pressing, M = oil after storage in a tank, N = oil after filling the final bottle

S/N – Signal/Noise Ratio, n.d. - not detectable (S/N < 3), n.q. – not quanitifable (S/N < 10)

RI – Retention Index

RI (HP5) – RI as determined in the measurements

RI Lit – referenced RI obtained from databases

[1] – RI obtained from http://www.flavornet.org

[2] – RI obtained from http://webbook.nist.gov/

[3] - Odour description obtained from http://www.thegoodscentscompany.com/

[t] - Tentatively identified, tentative identification is based on the mass spectra and RIs obtained from RI databases

[*] - OAV determined experimentally

Odour thresholds were collected from "Odour thresholds: compilations of odour threshold values in air, water and other media" – L.J. van Gemert, using the matrix oil/vegetable oil

5.2.1.1. Sensory evaluation

Sensory analysis, in terms of QDA, of the raw and roasted hempseeds as well as the freshly pressed hempseed oil was performed by the sensory expert panel of Graz University of Technology. The analysis was done in the sensory lab of the Institute of Analytical Chemistry and Food Chemistry under standardized conditions. Data acquisition was performed via Compusense software on tablet computers. The attributes were chosen according to the previously determined volatile compounds of the samples and the obtained odour descriptions. Furthermore, at the beginning of this study, a descriptive analysis of all three oils has been performed with the sensory expert panel of Graz University of Technology. This first evaluation also gave a basic impression of the odour and taste of the oils investigated. The spiderweb plot (Figure 44) shows significant differences in the odour of the investigated samples. The panellists described the odour in the raw material as more vegetable-like and green/grassy, as the roasting progressed the odour developed into a nuttier and roasted smell. The final oil was described as nutty and roasty, but with a note of vegetable-like and green/grassy odour. Overall, the raw material of the hempseeds and the hempseeds after 10 minutes of roasting were rated as mostly green/grassy and vegetable-like. With an increased roasting time, also the frequency of the attributes nutty and roasty increased and were still strongly perceived in the pressed oil. In the final product, on the other hand, the attributes green/grassy and vegetable-like smell decreased.

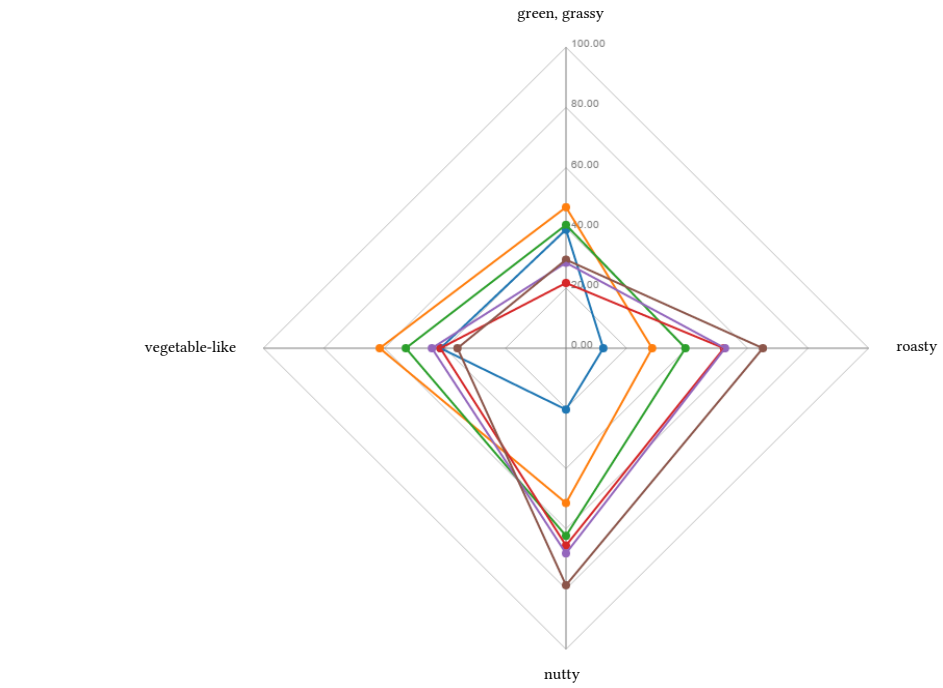


Figure 44: Spiderweb plot showing the results of the QDA different samples of the roasting process of hempseeds and the freshly pressed oil; values are presented as mean values from individual intensity score (n=9)

When looking at the respective PCA (Figure 45) the results are presented in a different manner. The green/grassy and vegetable like odour impressions are positioned near the samples after 10 and 20 minutes of roasting and therefore show a correlation (see Quadrant III). The nutty and roasty odour impressions, on the contrary, are positioned near the samples after 30 and 40 minutes of roasting and the freshly pressed hempseed oil and imply a correlation of the theses samples with the nutty and roasty attributes (see Quadrants I and IV). As seen in the PCA, the raw material is positioned in a different quadrant (see Quadrant II) and therefore shows no correlation to the other roasted samples.

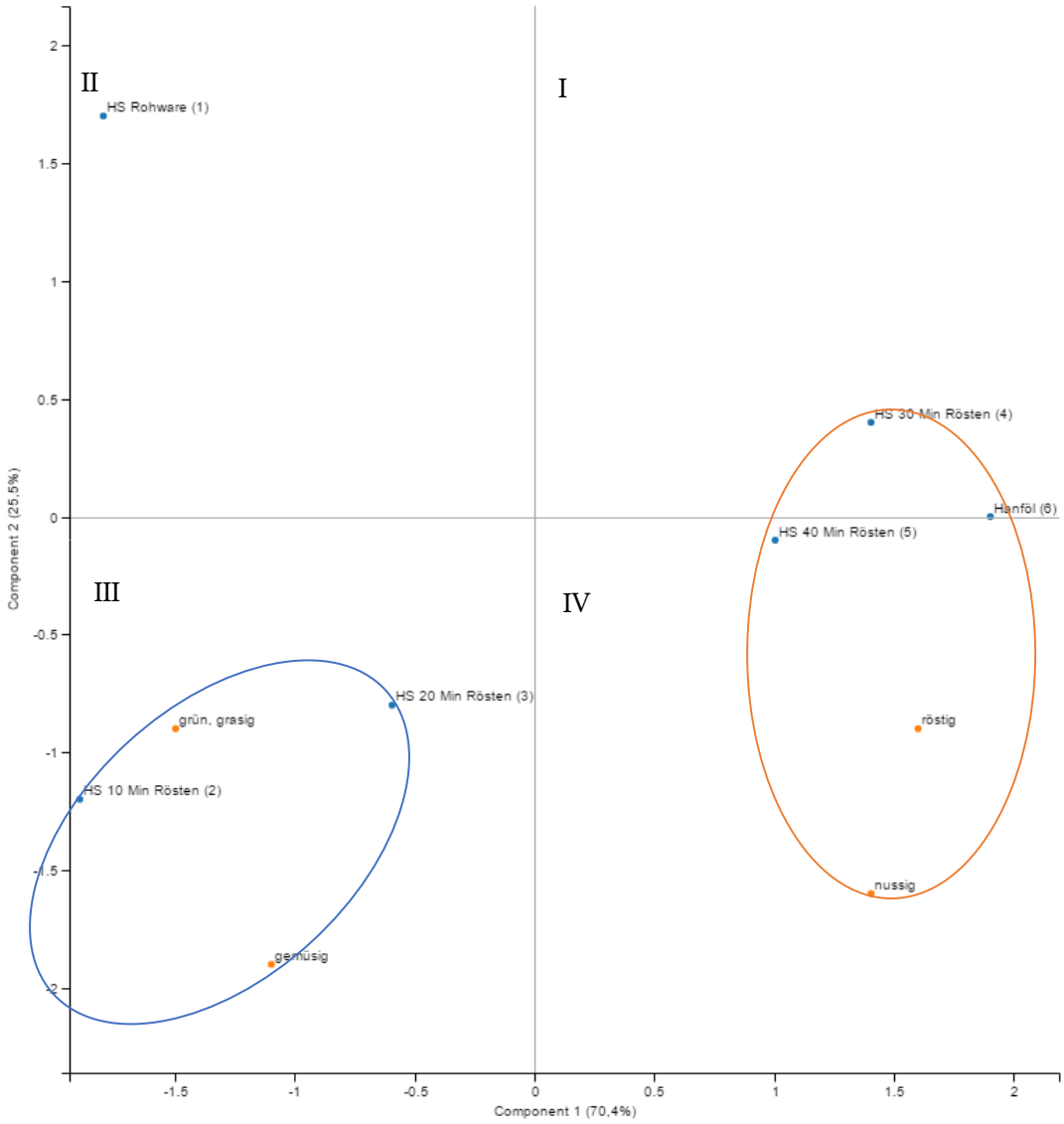
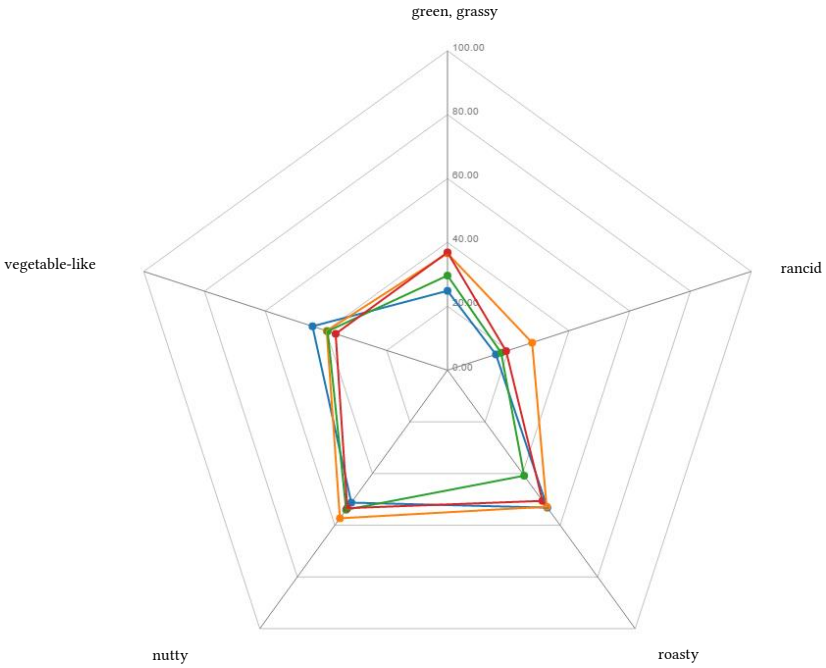


Figure 45: PCA showing the results of different samples from the roasting process of hempseed oil

The odour of the hempseed oil was investigated over the different stages of storage after the pressing of the oil. The first sample was taken immediately after the pressing, the second sample one day after the pressing. The oil was then stored in tanks and a sample was taken before the final filling of the oil. The last sample was taken after filling the oil into flasks. The panellists could not determine statistically significant differences in those samples. These results are shown in the spiderweb in Figure 46 as well as in the PCA in Figure 47.

The investigated attributes green/grassy, vegetable-like, nutty, roasty and rancid odour did not differ for the samples analysed. A weak rancid note was observed for the oil that has been in storage for one day, however, no statistically significant difference in rancidity in the different samples could be detected.



Blue: pressed oil
Orange: one day after pressing of the oil
Green: after storage in the tank
Red: filled oil

Figure 46: Spiderweb plot showing the results of the QDA different samples of the storage process of hempseeds and the freshly pressed oil; values are presented as mean values from individual intensity score (n=12)

The analysis of the obtained sensory evaluation of the different storage stages of the hempseed oil shows some differences in the given PCA in Figure 47. As highlighted, the freshly pressed hempseed oil was associated with a vegetable-like smell (see Quadrant IV). The filled hempseed oil was associated with a green/grassy and nutty odour but is also in the periphery of the oil sample one day after pressing, which was associated with a rancid odour (see Quadrants II and III). The hempseed oil from the storage tank shows no correlation to the other samples (see Quadrant I). As mentioned above, the values obtained from QDA were not statistically significant, so ultimately no sound difference could be measured.

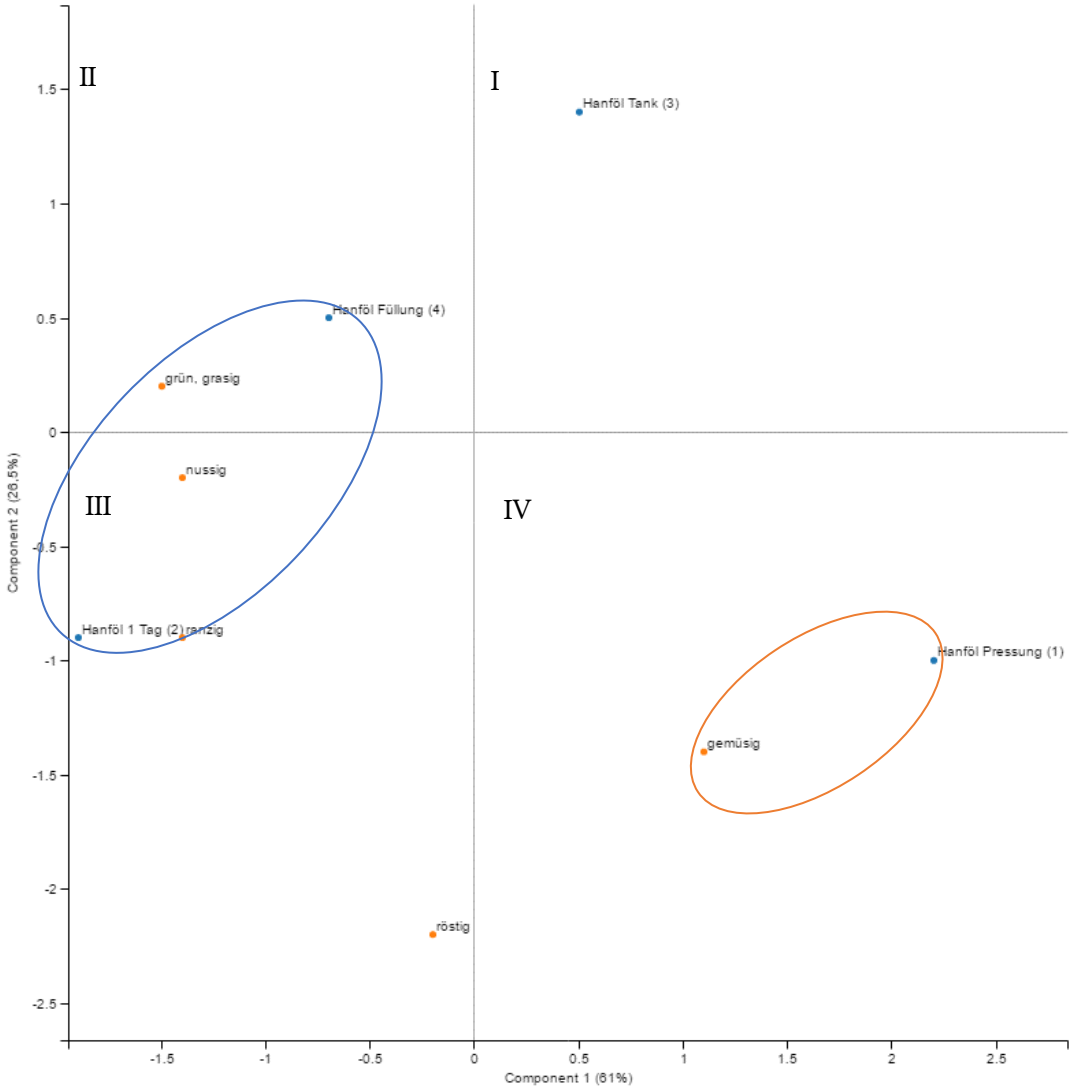


Figure 47: PCA showing the results of different samples from the storage process of hempseed oil

5.2.2. Comparison of bottled oil with different BBD

The chia seed, flaxseed and hempseed oils were investigated using HS SPME GC-MS to identify and quantify the volatile compounds. In Figure 48,Figure 49 andFigure 50 the GC-MS chromatograms obtained from these investigations can be seen. In all three figures, the chromatograms are stacked starting with the least durable oil on top continuing to the longest durable oil at the bottom of the figure. The determined compounds with the corresponding area and retention indices as well as the according odour description can be found in Table 19 for the investigated chia seed oil, in Table 20 for the investigated flaxseed oil and in Table 21 for the investigated hempseed oil. In all investigated oils, carbonyl compounds, like 2-methyl-butanal, 3-methyl-butanal or benzaldehyde, could be found. These are also known as Strecker aldehydes and are most likely derived from the amino acid precursor's valine, isoleucine, leucine and alanine. Intense thermal treatment is required to generate these aldehydes as they were only reported in oil samples with a roasting time of over 20-30 minutes [65].

In all samples α-pinene could be detected, this compound is found in fennel, coriander or in rosemary, but has been reported to be found in a variety of oils as well. The flavour has also been described as turpentine and pine-like [68].

The determination of acetic acid was expected, as this acid is formed while processing the seeds [60].

In all samples propanoic acid as well as 1-methoxy-2-propanal, whereas hexanal and 2-hexenal could be found in the chia seed oil samples. Propanal is believed to oxidize to propanoic acid and exceeds as a product of oxidation. Also, propanal and hexanal can be formed as the oxidation process continues [61]. Additionally, oxidation indicator compounds, like 2-ethyl-furan, hexanal and 2-pentenal could be detected, which are found to produce a rancid off-flavour, for example, in olive oil [69].
In the investigated **chia seed oil** samples 2 acids, 7 alcohols, 8 aldehydes, 2 furan derivatives, 2 ketones, 8 terpenes and 1 other compound were identified. Furthermore, camphene could be detected in chia seed and hempseed oil, which is a terpene found in a variety of essential oils, such as turpentine, cypress or bergamot oil. Also, essential oil derived from nutmeg contains 60-80% of camphene [70]. In chia seed oil and flaxseed oil 1-penten-3-ol was found, this can be used as a food additive to enhance buttery and green flavours [71]. In the samples 2-methyl-1-butanol was found, which is said to have a malt-like flavour [68].

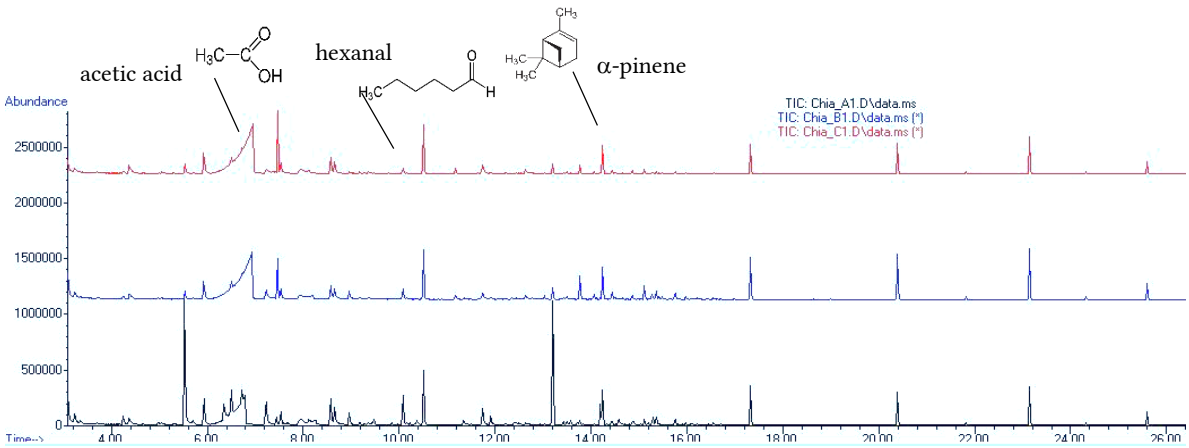


Figure 48: Stacked GC-MS chromatograms of the three investigated bottled organic chia seed oil analysed on the June 20, 2017 (BBD from top to bottom: March 01, 2017 (red), November 28, 2017 (blue) and May 15, 2018 (black)), vertical axis showing the abundance in peak area, horizontal axis showing the time in minutes

In the **flaxseed oil** samples 1 acid, 14 alcohols, 6 aldehydes, 2 furan derivatives, 2 ketones, 11 terpenes and 1 other compound could be identified. δ-3-carene could be identified in flaxseed and hempseed oil and is found to be giving turpentine like flavour [72]. The found 1-hexanol in flaxseed and hempseed oil can be used to enhance coconut and berry flavours and is overall described as fruity [73]. D-limonene was found in flaxseed and hempseed oil, which is a cyclic terpene. The d-isomer shows a strong smell of oranges, the l-isomer, on the other hand presents a piney, turpentine-like odour [57]. In the samples 2-methyl-1-butanol was found in flaxseed oil, which has a flavour that is described as malty [68].

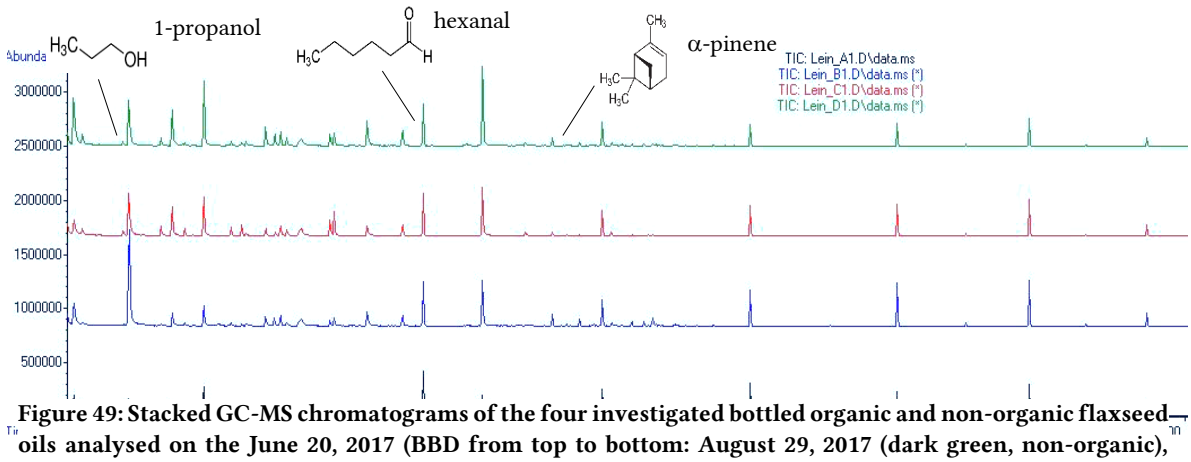


Figure 49: Stacked GC-MS chromatograms of the four investigated bottled organic and non-organic flaxseed oils analysed on the June 20, 2017 (BBD from top to bottom: August 29, 2017 (dark green, non-organic), September 25, 2017 (red, organic), May 27, 2017 (blue, non-organic) and May 27, 2017 (black, organic), vertical axis showing the abundance in peak area, horizontal axis showing the time in minutes

Overall, in the **hempseed oil** samples 6 acids, 7 alcohols, 15 aldehydes, 2 furan derivatives, 2 ketones, 11 terpenes, 5 alkenes, 8 pyrazines and 2 other compounds could be identified. In the hempseed oil volatiles like methyl-pyrazine, 2,5-dimethyl-pyrazine, trimethyl-pyrazine, 3-ethyl-2,5-dimethyl-pyrazine could be identified. N-heterolytic compounds like pyrazines are formed at the carbonyl-amine condensation of two amino ketones. These are usually generated during the Strecker reaction. Zhang *et al.* [65] reported in 2016 that pyrazines could only be found in roasted palm kernel oil and are absent in unroasted palm kernel oil. Also, other reports show the same findings. This indicates that pyrazines are generated upon heating, as Van Boekel *et. al.* found in 2006 that pyrazines in foods are formed over a thermal processing of over 100°C [65]. Siegmund and Murkovic [66] demonstrated in 2004 that high temperatures in the roasting process are necessary to form the typical nutty and roasty aroma in pumpkin seed oil. In this study compounds such as alkylated pyrazines could be determined for the typical nutty and roasty flavour of this oil, which need a temperature over 90°C for their formation [66]. Additionally, it was found that the amount of pyrazines in different types of oils is sensitive to the degree of unsaturated fatty acids in the oil [74]. 2-Octene was detected, which, like 1-octene, has been reported to be, a product from lipid oxidation decomposition [75]. Furthermore, β-Myrcene could be detected in the hempseed oil samples, which is found to be olefinic natural organic hydrocarbon that can be found in many plants as a component of essential oils [76].

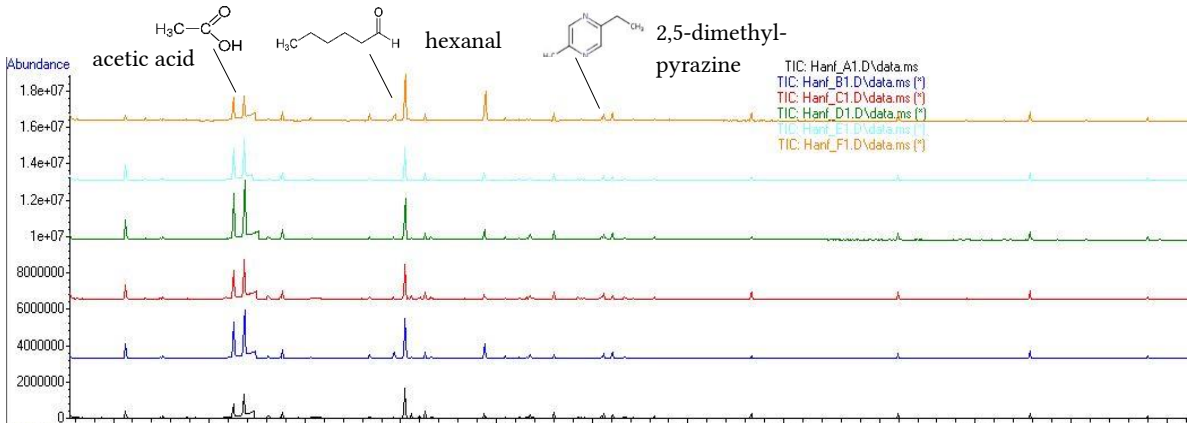


Figure 50: Stacked GC-MS chromatograms of the three investigated bottled hempseed oils analysed on June 28, 2017 (BBD from top to bottom: May 03, 2018 (orange, non-organic), May 03, 2018 (light blue, organic), October 27, 2017 (dark green, non-organic), November 03, 2017 (red, organic), April 01, 2017 (blue, non-organic) and April 01, 2017 (black, organic)), vertical axis showing the abundance in peak area, horizontal axis showing the time in minutes

Table 19: Volatile compounds identified in organic chia seed oil (measured on 20.06.2017); values are given in terms of mean areas from HS SPME GC-MS (n=4). calculated RI value in comparison to the RI Lit values found in literature

Compound	RI (HP5)	RI Lit	Odour description	Area (01.03.2017)	Area (28.11.2017)	Area (15.05.2018)
Acids						
Acetic acid	620	600[1]	sour	20863094	2182463	1959203
Propanoic acid[t]	723		acidic, dairy, fruity[3]	8433441	5181126	2945156
Alcohols						
2-Butanol	611	612[2]	sweet, apricot[3]	n.d.	314015	228708
1-Penten-3-ol	688	686[1]	butter, pungent	5671464	2402943	21601464
3-Methyl-1-butanol	741	736[1]	whiskey, malt, burnt	4885044	2625488	3933485
2-Methyl-1-butanol	744	739[1]	malt	3852781	2625488	2950731
1-Pentanol	772	766[1]	balsamic	n.d.	539786	456990
1-Hexanol	871	851[1]	resin, flower, green	2515901	1356359 n.d.	
Aldehydes						
2-Methyl-1-propanal	636	554[2]	fusel, whiskey[3]	4891362	n.d.	n.d.
2-Butenal[t]	653			8479785	n.d.	n.d.
3-Methyl-butanal	659	650[1]	malt	10145779	n.d.	n.d.
2-Pentenal	756	754[1]	strawberry, fruit, tomato	2145507	1736419	1618921
Hexanal	801	801[1]	grass, tallow, fat	5392213	2193104	888581
2-Hexenal	854	854[1]	apple, green	1146979	427653	567755
2-Heptenal	959	957[1]	soap, fat, almond	911716	n.d.	n.d.
2,4-Heptadienal	996	1000[1]	fried	1729852	n.d.	n.d.
Alkanes						
Undecane	1100	1100[1]	alkane	218145	n.d.	n.d.
Decane	1000	1000[1]	alkane	n.d.	n.d.	163976
Alkene						
Ethyl-benzene	860	853[2]		n.d.	110050	91018
3,5-Octadiene[t]	813			943144	n.d.	n.d.
Furan derivatives				n.d.	n.d.	n.d.
Tetrahydro-furan	699	701[2]		n.d.	n.d.	227011
2-Ethyl-furan	700	701[2]	solvent, dirty, musty, brown, earthy[3]	2099584	1847911	4797211
Ketones						
4-Methyl-2-hexanone[t]	847			n.d.	921088	n.d.
3,5-Octadien-2-one	1070	1052[2]	fatty, fruity, hay, green, herbal[3]	546375	278753	n.d.
Terpenes						

Compound	RI (HP5)	RI Lit	Odour description	Area (01.03.2017)	Area (28.11.2017)	Area (15.05.2018)
Styrene	891	893[1]	baslsamic, gasoline	384173	n.d.	286946
α-Thujene	927	938[1]	wood, green, herb	261411	n.d.	n.d.
α-Pinene	934	939[1]	pine, turpentine	20181257	1955915	1674950
Camphene	951	953[1]	camphor	2207929	512817	92689
2-β-Pinene	979	981[1]	pine, resin, turpentine	3242602	n.d.	133409
α-Terpinene	1018	1012[1]	lemon	151162	n.d.	n.d.
D-Limonene	1031	1030[1]	citrus, mint	1505513	n.d.	n.d.
γ-Terpinene	1057	1074[1]	gasloine, turpentine	793355	n.d.	n.d.
α-Terpinolene[t]	1087		woody, terpenic, lemon, lime, herbal, floral[3]	199302	n.d.	n.d.
δ-3-Carene	1010	1009[1]	lemon, resin	n.d.	918486	543167
Other compounds						
γ -Butyrolacetone	909	908[2]	creamy, oily, fatty[3]	731439	1370579	1276587

Results are expressed as average area values calculated from the determined areas n=4, n.d. – not detected

RI – Retention index

RI (HP5) – RI experimentally determined

RI Lit – referenced RI obtained from databases

[1] – RI obtained from http://www.flavornet.org

[2] – RI obtained from http://webbook.nist.gov/

[3] - Odour description obtained from http://www.thegoodscentscompany.com/

[t] - Tentatively identified, tentative identification is based on the mass spectra and RIs obtained from RI databases

Table 20: Volatile compounds identified in organic and non-organic flaxseed oil (measured on the 20.06.2017); values are given in terms of average areas from HS SPME GC-MS (n=4), calculated RI value in comparison to the RI Lit values found in literature

Compound	RI (HP5)	RI Lit	Odour description	Area^{O} (27.05.2017)	Area (27.05.2017)	Area^{O} (05.09.2017)	Area (05.09.2017)
Acids							
Acetic acid	620	600[1]	sour	5476862	5913968	3941538	12852933
Alcohols							
1-Hexanol	871	851[1]	resin, flower, green	n.d.	27154134	n.d.	n.d.
2-Butanol	610	612[2]	sweet, apricot[3]	1957679	714629	11292063	11887870
2-Methyl-3-buten-2-ol^{t}	618		herbal, earthy, oily[3]	n.d.	n.d.	n.d.	1335070
1-Butanol	671	675[1]	medicine, fruit		3546501	5845680	177733
1-Penten-3-ol	688	686[1]	butter, pungent	269694	386777	1244307	613221
3-Methyl-1-butanol	741	736[1]	whiskey, malt, burnt	5442414	271660	1745250	13030830
2-Methyl-1-butanol	744	739[1]	malt	381409	554061	517454	827114
1-Pentanol	771	766[1]	fruit	284806	1719555	1540011	670381
3-Hexen-1-ol	857	858[1]	grass	n.d.	n.d.	591880	n.d.
2-Hexen-1-ol	867	854[2]	sweet, fruity, green, fatty[3]	n.d.	n.d.	n.d.	178912
1-Hexanol	870	851[1]	resin, flower, green	1285195	n.d.	1561265	910748
2-Heptanol	902	904[2]	fruity, green, earthy, bitter[3]	n.d.	n.d.	n.d.	1372814
Eucalyptol	1035	1033[2]	eucalyptus, herbal, camphor, medicinal[3]	n.d.	n.d.	265008	3618188
Aldehydes							
3-Methyl-butanal	659	650[1]	malt	506380	1452999	1115470	1920386
2-Methyl-butanal	668	641[1]	cocoa, almond	842595	999946	2920657	2401081
Hexanal	801	801[1]	grass, tallow, fat	1249024	3006829	4602102	4948707
2-Hexenal	854	854[1]	apple, green	n.d.	n.d.	n.d.	3247650
Benzaldehyde	1132	960[1]	almond, burnt sugar	n.d.	2028546	2454419	516421
Alkanes							
3-Methyl-hexane	675	667[2]		n.d.	n.d.	n.d.	371266
Decane	1000	1000[1]	alkane	n.d.	n.d.	n.d.	141084
Furan derivatives							
2-Ethyl-furan	700	701[2]	sweet, burnt, earthy, malty[3]	300397	9065605	10187772	194656
2-Pentyl-furan	990	989[2]	fruity, green, earthy, beany, vegetable[3]	n.d.	364476	n.d.	240373
Ketones							

Compound	RI (HP5)	RI Lit	Odour description	Area^{O} (27.05.2017)	Area (27.05.2017)	Area^{O} (05.09.2017)	Area (05.09.2017)
2-Butanone^{t}	600		etheral, fruity, camphoreous[3]	n.d.	n.d.	1129716	734776
2,3-Pentanedione^{t}	702		buttery, nutty, toasted, caramellic[3]	n.d.	n.d.	78234	n.d.
Terpenes							
α-Thujene	927	938[1]	wood, green herb	n.d.	n.d.	708793	n.d.
α-Pinene	934	939[1]	pine, turpentine	950534	738988	712489	101577
β-Myrcene	990	992[1]	balsamic, must, spice	n.d.	n.d.	36467	n.d.
Camphene	951	953[1]	camphor	n.d.	n.d.	n.d.	499018
Phellandrene	1007	1007[1]	turpentine, mint, spice	n.d.	230841	n.d.	
δ-3-Carene	1010	1009[1]	lemon, resin	186396	841754	242057	220207
p-Cymene	1026	1027[1]	solvent, gasoline, citrus	284479	n.d.	216538	n.d.
D-Limonene	1031	1030[1]	citrus, mint	521963	1792390	146972	314686
γ-Terpinene	1060	1074[1]	gasoline, turpentine	n.d.	n.d.	94027	n.d.
β-Thujone^{t}	1109		cedar, thujonic, spicy, woody[3]	n.d.	1109	55660	n.d.
Styrene	891	893[1]	baslsamic, gasoline	n.d.	n.d.	n.d.	1084540
Other compounds							
γ-Butyrolacetone	909	908[2]	creamy, oily, fatty[3]	4923600	4278851	4923600	3215624

Results are expressed as average area values calculated from the determined areas n=4, n.d. – not detected

Area^{O} – indicates organic oil samples

RI – Retention index

RI (HP5) – RI experimentally determined

RI Lit – referencened RI obtained from databases

[1] – RI obtained from http://www.flavornet.org

[2] – RI obtained from http://webbook.nist.gov/

[3] - Odour description obtained from http://www.thegoodscentscompany.com/

^{t} - Tentatively identified, tentative identification is based on the mass spectra and RIs obtained from RI databases

Table 21: Volatile compounds identified in organic and non-organic hempseed oil (measured on the 28.06.2017); values are given in terms of average areas from HS SPME GC-MS (n=4), calculated RI value in comparison to the RI Lit values found in literature

Compound	RI (HP5)	RI Lit	Odour description	Area^{O} (01.04.2017)	Area (01.04.2017)	Area^{O} (03.11.2017)	Area (27.10.2017)	Area^{O} (03.05.2018)	Area (03.05.2018)
Acids									
Acetic acid	606	600[1]	sour	33374882	36035404	36623875	26269877	14626501	37677302
Butanoic acid^{t}	791			n.d.	n.d.	n.d.	n.d.	1041401	656593
Hexanoic acid^{t}	984		sour, fatty, sweaty, cheesy[3]	n.d.	n.d.	n.d.	2306102	n.d.	494031
2-Methyl-2-propenoic acid^{t}	713			637110	n.d.	n.d.	n.d.	n.d.	n.d.
Propanoic acid	728	668[1]	pungent, rancid, soy	5138105	7365580	4807166	n.d.	n.d.	n.d.
Pentanoic acid	887		acidic, sharp, cheesy, sour, milky, tobacco, fruity[3]	n.d.	670506	n.d.	n.d.	n.d.	n.d.
Alcohols									
1-Penten-3-ol	688	686[1]	butter, pungent	4595744	n.d.	5411561	5098901	n.d.	n.d.
1-Pentanol	772	766[1]	balsamic	2623162	6077765	2648419	3044140	2716871	8997416
1-Hexanol	871	851[1]	resin, fruit, green	6430367	18260015	5869509	11359153	8392712	41625486
1-Heptanol	970	962[1]	chemical, green		n.d.	n.d.	n.d.	n.d.	718297
2,3-Butanediol	793	806[1]	fruit, onion	n.d.	10872185	n.d.	2633662	n.d.	13145062
3-Methyl-1-butanol	741	736[1]	whiskey, malt, burnt		n.d.	n.d.	n.d.	n.d.	1614670
Aldehydes									
Butanal	586	596[1]	pungent, green	1317344	1629615	1671828	2059468	1197904	1954190
3-Methyl-butanal	659	650[1]	malt	21711375	48080323	31319118	58912035	32529348	30514291
2-Methyl-butanal	668	641[1]	cocoa, almond	39312582	67466088	54227696	81650473	49626599	41130118
Pentanal	700	732[1]	almond, malt, pungent	7606147	9537664	8394993	12204278	7286510	8348760
2-Pentenal	756	754[1]	strawberry, fruit, tomato		n.d.	n.d.	n.d.	n.d.	514146
2-Methyl-2-butenal	743	753[1]	green, fruit	n.d.	1272632	n.d.	n.d.	n.d.	n.d.
Hexanal	801	801[1]	grass, tallow, fat	34044371	47825388	35486100	45388671	32302055	56424528
2-Hexenal	854	854[1]	apple, green	914952	907281	1214779	1271617	735650	1266312
2-Heptenal	957	957[1]	soap, fat, almond	1372842	n.d.	2578623	658863	950048	581632
Heptanal	902	903[1]	fat, citrus, rancid		1825086	659132	1828114	n.d.	2445404
Furfural	833	829[1]	bread, almond, sweet		n.d.	n.d.	n.d.	455837	n.d.
Benzaldehyde	963	961[1]	almond, burnt sugar	619746	993126	1022559	1183147	731303	701661
Decanal^{t}	1022		sweet, aldehydic, orange, waxy, citrus rind[3]	n.d.	n.d.	n.d.	632921	n.d.	n.d.
Nonanal	1105	1104[1]	fat, citrus, green		468468	234302	594690	297605	428681
2-Octenal	1059	1049[1]	green leaf, walnut		n.d.	n.d.	n.d.	265891	n.d.
Alkanes									

Compound	RI (HP5)	RI Lit	Odour description	Area^{O} (01.04.2017)	Area (01.04.2017)	Area^{O} (03.11.2017)	Area (27.10.2017)	Area^{O} (03.05.2018)	Area (03.05.2018)
Dodecane	960		alkane	434685	n.d.	n.d.	n.d.	n.d.	n.d.
Undecane	1100	1100[1]	alkane	n.d.	n.d.	n.d.	n.d.	n.d.	148018
Alkenes									
1-Octene	791	790[2]		1339645	n.d.	1328579	n.d.	n.d.	n.d.
2-Octene	814	813[2]		3087797	1165604	2515051	443755	430510	430505
1-Methyl-2-benzene^{t}	1026			n.d.	829577	n.d.	n.d.	n.d.	n.d.
Methyl-benzene^{t}	1092			n.d.	1588097	n.d.	n.d.	n.d.	n.d.
Furan derivative									
2,3-Dihydro-furan^{t}	653			n.d.	n.d.	690182	n.d.	n.d.	n.d.
2(3H)-Furanone	1051	1049[1]	caramel, sweet, milde		n.d.	n.d.	n.d.	n.d.	1795555
Ketones									
2-Butanone	600	602[2]	ethereal, fruity, camphoreous[3]	n.d.	2784716	2737233	3331648	1822923	n.d.
2-Hexanone	789	792[1]	ether	n.d.	n.d.	n.d.	n.d.	245484	n.d.
2-Heptanone	889	895[1]	soap	1740061	2655542	1583587	3447371	1394433	3592377
3,5-Octadien-2-one	1069	1052[1]	fatty, fruity, hay, green, herbal[3]	n.d.	828748	n.d.	n.d.	n.d.	1251260
3-Octen-2-one	1038	1040[1]	nut, crsuhed bug		n.d.	n.d.	433734	n.d.	n.d.
2-Pentanone	688	711[2]	ether, fruit	n.d.	n.d.	n.d.	n.d.	n.d.	2770140
Terpenes									
α-Thujene	927	938[1]	wood, green, herb		n.d.	n.d.	n.d.	n.d.	509674
α-Pinene	934	939[1]	pine, turpentine	6462481	4185072	6860677	8363945	5661566	7054211
Camphene	951	953[1]	camphor	1081718	880562	1025594	1074208	849459	1146530
2-β-Pinene	980	981[1]	pine, resin, turpentine		n.d.	2834973	2923192	1278014	2871483
β-Mycrene	989	992[1]	blasmamic, must, spice	4436457	6934269	3822470	6790428	2945441	4680612
β-Ocimene	1047	1043[1]	citrus, herb, flower		n.d.	n.d.	n.d.	n.d.	1237297
δ-3-Carene	1010	1009[1]	lemon, resin	1214477	1272696	894451	766888	839440	2229550
α-Terpinene	1018	1012[1]	lemon	n.d.	n.d.	n.d.	n.d.	n.d.	225911
p-Cymene	1026	1027[1]	solvent, gasoline, citrus		n.d.	n.d.	n.d.	398792	1147553
D-Limonene	1031	1030[1]	citurs, mint	3695571	2803601	1704631	3282274	2232195	2239500
γ-Terpinene	1060	1074[1]	gasoline, turpentine		314323	n.d.	n.d.	n.d.	n.d.
α-Terpinolene	1087	1090[1]	pine, plastic		965929	n.d.	n.d.	309687	1253939
Other compounds									
γ-Butyrolactone	909	908[2]	creamy, oily, fatty	n.d.	1733093	1784322	2202074	996400	1212603
Pyrazines									
Methyl-pyrazine	824	828[1]	popcorn	2410362	3440257	2970302	3799995	1828074	877750
2,5-Dimethyl-pyrazine	912	905[1]	cocoa, roasted nut, roasted beef, medicine	6603029	4943946	4593888	6696526	2872078	2295787

Compound	RI (HP5)	RI Lit	Odour description	Area^{O} (01.04.2017)	Area (01.04.2017)	Area^{O} (03.11.2017)	Area (27.10.2017)	Area^{O} (03.05.2018)	Area (03.05.2018)
Ethyl-pyrazine	915	907[1]	peanut butter, wood		n.d.	567816	n.d.	n.d.	n.d.
2,3-Dimethyl-pyrazine	917	913[1]	roasted nut, cocoa, roast beef			476012	n.d.	n.d.	n.d.
2-Ethyl-6-methyl-pyrazine	997	993[1]	fruit, sweet		n.d.	318870	355326	n.d.	n.d.
3-Ethyl-2,5-dimethyl-pyrazine	1076	1082[1]	potato, roast		648826	520741	947409	299899	n.d.
2-Ethyl-3,5-dimethyl-pyrazine	1076	1082[1]	potato, roast		n.d.	n.d.	n.d.	n.d.	318170
Trimethyl-pyrazine	1001	1000[1]	roast, potato, must	2324369	n.d.	2524928	n.d.	1662604	1083949

Results are expressed as average area values calculated from the determined areas n=4, n.d.- not detected

Area^{O} – indicates organic oil samples

RI – Retention index

RI (HP5) – RI experimentally determined

RI Lit – referencened RI obtained from databases

[1] – RI obtained from http://www.flavornet.org

[2] – RI obtained from http://webbook.nist.gov/

[3] - Odour description obtained from http://www.thegoodscentscompany.com/

[t]- Tentatively identified, tentative identification is based on the mass spectra and RIs obtained from RI databases

5.2.2.1. Fatty acid composition

The investigated chia seed oils showed no significant changes in the fatty acid composition over time. A value of 62-64% of α-linolenic acid could be detected in the three samples. It should also be mentioned that α-linolenic acid (C18:3) is the fatty acid that makes the biggest part of the fatty acid profile in the oil samples of the chia seed oil, as seen from Table 22. α-linolenic acid has been reported to be the main fatty acid in chia seeds and chia seed oil. Ixtaina, *et al.* [77] reported in 2010 an α-linolenic acid value of 52.8% and 64.8% in two different kinds of chia seed oils. Ciftci, Przybylski, & Rudzinska [78] reported in 2012 an α-linoleic acid value of approx. 59.8% in chia seeds. The four-tested flaxseed oil also showed no significant differences, 52-56% of α-linolenic acid could be detected as well as 15% of linoleic acid and 16-20% of oleic acid, as seen from the tested flaxseed oils showed no significant differences between the four investigated samples. Krist *et al.* [22] reported in 2006 45-55% of α-linolenic acid, 16-20% of linoleic acid and 17-24% oleic acid in flaxseed oil. 52-56% of linoleic acid, 16-18% of α-linolenic acid as well as 1-3% of ɣ-linolenic acid and 10-13% of oleic acid were found in the hempseed oil samples, as seen from Table 24 and Table 25. Parker *et al.* [23] reported in 2003 a fatty acid composition of hempseed oil of approx. 60% linoleic acid, 19% α-linolenic acid and 11% oleic acid. Also, ɣ-linolenic acid of around 2-4% has been reported for hempseed oil [8]. A comparison of the fatty acid composition of the investigated types of oil can be seen from Figure 51.

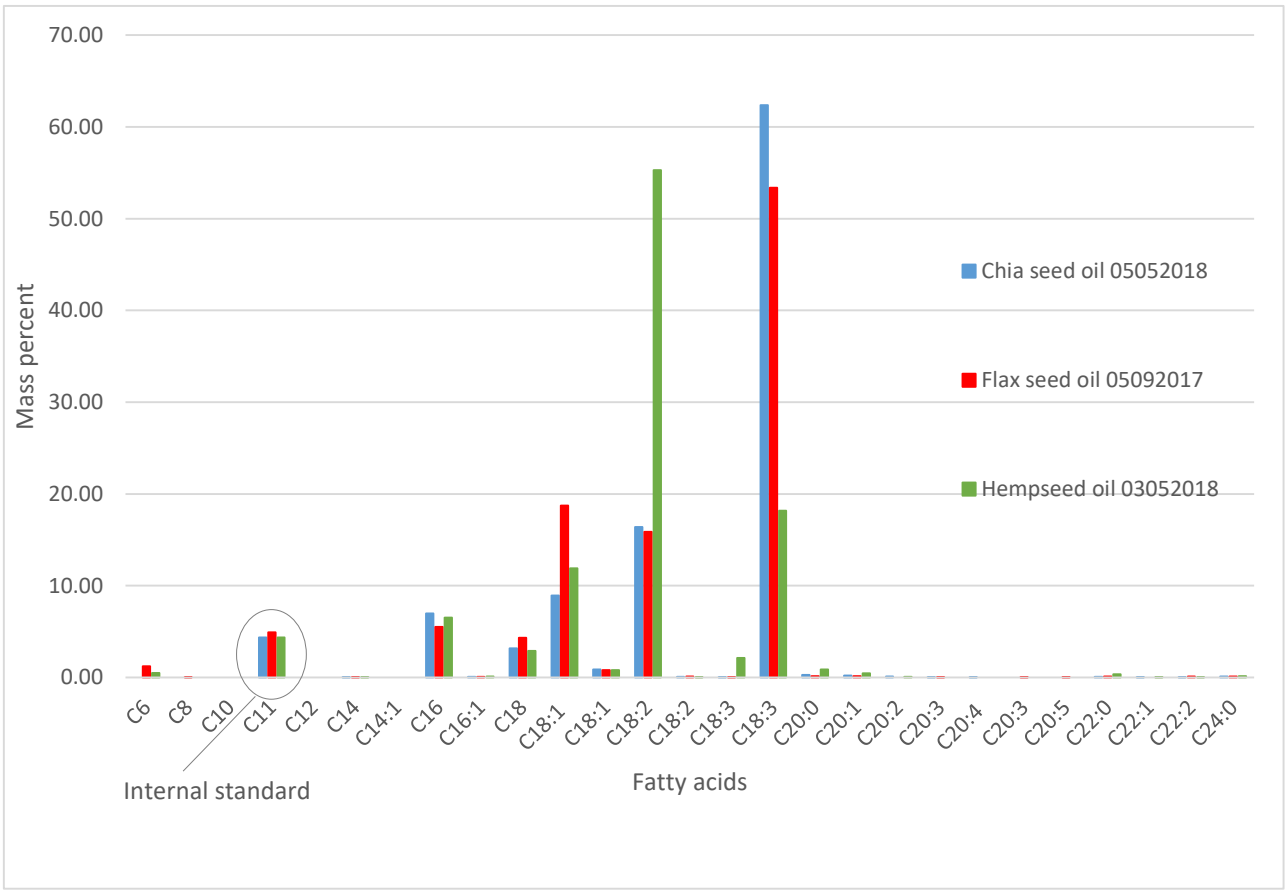


Figure 51: Comparison of the obtained fatty acid composition of chia seed oil, flaxseed oil and hempseed oil respectively values given as relative mass percent, C11 was used as an internal standard

Table 22: Fatty acid composition of the tested chia seed oils, given in g/kg and the percentage (n=4)

		Chia seed oil 01.03.2017	Chia seed oil 28.11.2017	Chia seed oil 15.05.2018	Chia seed oil 01.03.2017	Chia seed oil 28.11.2017	Chia seed oil 15.05.2018
	Fatty acids	**g/kg**	**g/kg**	**g/kg**	**%**	**%**	**%**
C6	Caproic acid	n.d.	n.d	n.d	n.d	n.d	n.d
C8	Caprylic acid	n.d	n.d	n.d	n.d	n.d	n.d
C10	Capric acid	n.d	n.d	n.d	n.d	n.d	n.d
C11 - IS	*Undecylic acid*	*37.22*	*32.71*	*36.11*	*4.67*	*3.45*	*4.35*
C12	Lauric acid	n.d	n.d	n.d	n.d	n.d	n.d
C14	Myristic acid	0.35	0.35	0.31	0.04	0.04	0.04
C14:1 ***(9Z)***	Myristoleic acid	n.d	n.d	n.d	n.d	n.d	n.d
C16	Palmitic acid	54.43	66.62	57.88	6.83	7.03	6.97
C16:1 ***(9Z)***	Palmitoleic acid	0.67	0.67	0.65	0.08	0.07	0.08
C18	Stearic acid	23.99	30.66	26.35	3.01	3.23	3.17
C18:1 ***(9Z)***	Oleic acid	43.80	63.68	74.26	5.49	6.72	8.94
C18:1 ***(9E)***	Elaidic acid	6.36	7.49	7.36	0.80	0.79	0.89
C18:2 ***(9Z, 12Z)***	Linoleic acid	160.75	156.02	136.21	20.16	16.46	16.40
C18:2 ***(9E, 12E)***	Linolelaidic acid	0.55	0.38	0.44	0.07	0.04	0.05
C18:3 ***(6Z, 9Z, 12Z)***	γ-Linolenic acid	n.d	0.55	0.32	n.d	0.06	0.04
C18:3 ***(9Z, 12Z, 15Z)***	α-Linolenic acid	495.89	611.74	518.13	62.18	64.53	62.37
C20:0	Eicosanic acid	1.55	2.44	2.25	0.19	0.26	0.27
C20:1 ***(11Z)***	cis-11-Eicosenic acid	1.58	1.48	1.60	0.20	0.16	0.19
C20:2 ***(11Z, 14Z)***	cis-11,14-Eicosadic acid	0.85	0.94	0.83	0.11	0.10	0.10
C20:3 ***(8Z, 11Z, 14Z)***	cis-8,11,14-Eicosatrienoic acid	n.d	n.d	n.d	n.d	n.d	n.d
C20:4 ***(5Z, 8Z, 11Z, 14Z)***	Arachidonic acid	0.38	0.48	0.31	0.05	0.05	0.04
C20:3 ***(11Z, 14Z, 17Z)***	cis-11,14,17-Eicosatrienoic acid	n.d	n.d	n.d	n.d	n.d	n.d
C20:5 ***(5Z, 8Z, 11Z, 14Z, 17Z)***	Eicosapentaenoic acid	0.73	n.d	n.d	0.09	n.d	n.d
C22:0	Behenic acid	0.45	0.67	0.69	0.06	0.07	0.08
C22:1 ***(13Z)***	Erucic acid	0.18	0.06	0.05	0.02	0.01	0.01
C22:2 ***(13Z, 16Z)***	cis-13,16-Docosadic acid	0.96	0.42	0.28	0.12	0.04	0.03
C24:0	Lignoceric acid	1.18	0.41	0.82	0.15	0.04	0.10

Results are expressed in percentage, as average calculated from four mesurments, n=4, n.d. – not detected
Additionally, results are expressed as g/kg, calculated with the used internal standard

Table 23: Fatty acid composition of the tested flaxseed oils, given in g/kg and the percentage (n=4)

		Flaxseed oil A 27.05.2017	Flaxseed oil B 27.05.2017	Flaxseed oil 05.09.2017	Flaxseed oil 29.08.2017	Flaxseed oil 27.05.2017	Flaxseed oil 27.05.2017	Flaxseed oil 05.09.2017	Flaxseed oil 29.08.2017
	Fatty acids	**g/kg**	**g/kg**	**g/kg**	**g/kg**	**%**	**%**	**%**	**%**
C6	Caproic acid	2.90	3.07	9.48	4.79	0.37	0.39	1.21	0.61
C8	Caprylic acid	n.d.	0.06	0.10	0.10	n.d.	0.01	0.01	0.01
C10	Capric acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C11 - IS	Undecylic acid	28.25	31.20	38.39	41.06	3.61	3.92	4.90	5.27
C12	Lauric acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C14	Myristic acid	0.34	0.33	0.19	0.24	0.04	0.04	0.02	0.03
C14:1 ***(9Z)***	Myristoleic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C16	Palmitic acid	41.78	41.24	43.20	40.18	5.34	5.18	5.52	5.16
C16:1 ***(9Z)***	Palmitoleic acid	0.58	0.49	0.67	0.50	0.07	0.06	0.09	0.06
C18	Stearic acid	35.64	31.44	33.73	30.18	4.56	3.95	4.31	3.87
C18:1 ***(9Z)***	Oleic acid	156.72	131.35	146.55	146.30	20.05	16.50	18.72	18.77
C18:1 ***(9E)***	Elaidic acid	5.60	5.39	6.17	5.24	0.72	0.68	0.79	0.67
C18:2 ***(9Z, 12Z)***	Linoleic acid	115.37	126.08	124.16	117.58	14.76	15.83	15.86	15.09
C18:2 ***(9E, 12E)***	Linolelaidic acid	0.93	0.68	0.87	0.79	0.12	0.09	0.11	0.10
C18:3 ***(6Z, 9Z, 12Z)***	γ-Linolenic acid	0.34	0.57	0.28	0.62	0.04	0.07	0.04	0.08
C18:3 ***(9Z, 12Z, 15Z)***	α-Linolenic acid	413.88	449.93	417.91	427.47	52.94	56.50	53.39	54.85
C20:0	Eicosanic acid	1.29	1.14	1.15	1.18	0.17	0.14	0.15	0.15
C20:1 ***(11Z)***	cis-11-Eicosenic acid	1.23	1.13	1.04	1.21	0.16	0.14	0.13	0.16
C20:2 ***(11Z, 14Z)***	cis-11,14-Eicosadic acid	0.30	0.27	n.d.	0.34	0.04	0.03	n.d.	0.04
C20:3 ***(8Z, 11Z, 14Z)***	cis-8,11,14-Eicosatrienoic acid	0.42	0.64	0.31	0.54	0.05	0.08	0.04	0.07
C20:4 ***(5Z, 8Z, 11Z, 14Z)***	Arachidonic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C20:3 ***(11Z, 14Z, 17Z)***	cis-11,14,17-Eicosatrienoic acid	0.43	0.53	0.25	0.53	0.06	0.07	0.03	0.07
C20:5 ***(5Z, 8Z, 11Z, 14Z, 17Z)***	Eicosapentaenoic acid	0.29	0.20	0.09	0.09	0.04	0.02	0.01	0.01
C22:0	Behenic acid	1.00	0.90	0.92	1.01	0.13	0.11	0.12	0.13
C22:1 ***(13Z)***	Erucic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C22:2 ***(13Z, 16Z)***	cis-13,16-Docosadic acid	0.67	0.43	0.75	0.55	0.09	0.05	0.10	0.07
C24:0	Lignoceric acid	0.82	0.70	0.73	0.72	0.10	0.09	0.09	0.09

Results are expressed in percentage, as average calculated from four mesurments, n=4, n.d. – not detected
Additionally, results are expressed as g/kg, calculated with the used internal standard

Table 24: Fatty acid composition of the tested hempseed oils given in g/kg (n=4)

		Hempseed oil A 01.04.2017	Hempseed oil B 01.04.2017	Hempseed oil 03.11.2017	Hempseed oil 27.10.2017	Hempseed oil E 03.05.2018	Hempseed oil F 03.05.2018
	Fatty acids	**g/kg**	**g/kg**	**g/kg**	**g/kg**	**g/kg**	**g/kg**
C6	Caproic acid	5.05	3.14	3.69	5.51	3.67	3.86
C8	Caprylic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C10	Capric acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C11 - IS	Undecylic acid	45.74	27.39	36.41	35.26	33.91	28.96
C12	Lauric acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C14	Myristic acid	n.d.	0.31	0.32	0.21	0.34	0.24
C14:1 ***(9Z)***	Myristoleic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C16	Palmitic acid	49.62	52.31	49.87	45.80	50.51	41.62
C16:1 ***(9Z)***	Palmitoleic acid	0.85	1.13	0.85	0.81	0.90	0.84
C18	Stearic acid	20.99	23.77	20.72	19.96	22.49	19.21
C18:1 ***(9Z)***	Oleic acid	89.37	104.59	89.36	89.62	92.14	69.89
C18:1 ***(9E)***	Elaidic acid	5.47	6.79	5.98	6.04	6.27	5.32
C18:2 ***(9Z, 12Z)***	Linoleic acid	416.90	439.74	415.55	382.27	428.62	377.75
C18:2 ***(9E, 12E)***	Linolelaidic acid	n.d.	0.40	0.28	0.68	0.40	0.34
C18:3 ***(6Z, 9Z, 12Z)***	γ-Linolenic acid	4.19	22.64	11.03	23.27	16.46	21.84
C18:3 ***(9Z, 12Z, 15Z)***	α-Linolenic acid	134.54	133.80	132.34	119.66	140.91	118.70
C20:0	Eicosanic acid	4.93	7.10	5.56	6.56	6.66	5.82
C20:1 ***(11Z)***	cis-11-Eicosenic acid	2.71	3.33	2.89	3.03	3.39	2.66
C20:2 ***(11Z, 14Z)***	cis-11,14-Eicosadic acid	0.56	0.58	0.60	0.58	0.59	0.48
C20:3 ***(8Z, 11Z, 14Z)***	cis-8,11,14-Eicosatrienoic acid	0.21	n.d.	n.d.	n.d.	n.d.	n.d.
C20:4 ***(5Z, 8Z, 11Z, 14Z)***	Arachidonic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C20:3 ***(11Z, 14Z, 17Z)***	cis-11,14,17-Eicosatrienoic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C20:5 ***(5Z, 8Z, 11Z, 14Z, 17Z)***	Eicosapentaenoic acid	n.d.	0.19	0.19	0.12	n.d.	0.06
C22:0	Behenic acid	1.77	2.69	2.18	2.75	2.57	2.10
C22:1 ***(13Z)***	Erucic acid	0.25	0.29	0.29	0.15	0.32	0.28
C22:2 ***(13Z, 16Z)***	cis-13,16-Docosadic acid	0.22	0.38	0.52	0.64	0.08	0.30
C24:0	Lignoceric acid	0.78	1.14	0.99	1.21	1.09	0.87

Results are expressed as g/kg, calculated with the used internal standard, n=4, n.d. – not detected

Table 25: Fatty acid composition of the tested hempseed oils given in percentage (n=4)

		Hempseed oil A 01.04.2017	Hempseed oil B 01.04.2017	Hempseed oil 03.11.2017	Hempseed oil 27.10.2017	Hempseed oil E 03.05.2018	Hempseed oil F 03.05.2018
	Fatty acids	**%**	**%**	**%**	**%**	**%**	**%**
C6	Caproic acid	0.64	0.39	0.50	0.78	0.47	0.58
C8	Caprylic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C10	Capric acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C11 - IS	Undecylic acid	5.81	3.41	4.90	5.00	4.37	4.32
C12	Lauric acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C14	Myristic acid	n.d.	0.04	0.04	0.03	0.04	0.04
C14:1 ***(9Z)***	Myristoleic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C16	Palmitic acid	6.30	6.51	6.71	6.49	6.52	6.21
C16:1 ***(9Z)***	Palmitoleic acid	0.11	0.14	0.11	0.12	0.12	0.13
C18	Stearic acid	2.66	2.96	2.79	2.83	2.90	2.87
C18:1 ***(9Z)***	Oleic acid	11.35	13.03	12.03	12.70	11.88	10.43
C18:1 ***(9E)***	Elaidic acid	0.69	0.85	0.81	0.86	0.81	0.79
C18:2 ***(9Z, 12Z)***	Linoleic acid	52.93	54.76	55.95	54.16	55.28	56.40
C18:2 ***(9E, 12E)***	Linolelaidic acid	n.d.	0.05	0.04	0.10	0.05	0.05
C18:3 ***(6Z, 9Z, 12Z)***	ɣ-Linolenic acid	0.53	2.82	1.48	3.30	2.12	3.26
C18:3 ***(9Z, 12Z, 15Z)***	α-Linolenic acid	17.08	16.66	17.82	16.95	18.17	17.72
C20:0	Eicosanic acid	0.63	0.88	0.75	0.93	0.86	0.87
C20:1 ***(11Z)***	cis-11-Eicosenic acid	0.34	0.41	0.39	0.43	0.44	0.40
C20:2 ***(11Z, 14Z)***	cis-11,14-Eicosadic acid	0.07	0.07	0.08	0.08	0.08	0.07
C20:3 ***(8Z, 11Z, 14Z)***	cis-8,11,14-Eicosatrienoic acid	0.03	n.d.	n.d.	n.d.	n.d.	n.d.
C20:4 ***(5Z, 8Z, 11Z, 14Z)***	Arachidonic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
C20:3 ***(11Z, 14Z, 17Z)***	cis-11,14,17-Eicosatrienoic acid	0.03	n.d.	n.d.	n.d.	n.d.	n.d.
C20:5 ***(5Z, 8Z, 11Z, 14Z, 17Z)***	Eicosapentaenoic acid	n.d.	0.02	0.03	0.02	0.00	0.01
C22:0	Behenic acid	0.22	0.34	0.29	0.39	0.33	0.31
C22:1 ***(13Z)***	Erucic acid	0.03	0.04	0.04	0.02	0.04	0.04
C22:2 ***(13Z, 16Z)***	cis-13,16-Docosadic acid	0.03	0.05	0.07	0.09	0.01	0.04
C24:0	Lignoceric acid	0.10	0.14	0.13	0.17	0.14	0.13

Results are expressed in percentage, as average calculated from four mesurments, n=4, n.d. – not detected

5.2.3. Sensory evaluation in den bottled oils

5.2.3.1. Evaluation of the sniffing sticks

After the identification of the volatile compounds of the bottled oils, those compounds that were supposed to have impact on the flavour of the oils and were of interest, were evaluated in their pure form by the expert panel. Adequate ethanolic solutions were prepared and applied on sniffing sticks. In Table 26 and Table 27 the compounds, followed by the found description of the odour in literature and the description of the panellist of the sensory expert panel of Graz University of Technology can be found. Again, these evaluations were done at the sensory lab of the Institute of Analytical Chemistry and Food Chemistry. The evaluation of the substances should help the members of the sensory test panel to train the typical odour impressions of the certain oils and to help gather a specific vocabulary for these impressions.

Table 26: Description of the substances tested with the sensory panel of the Graz University of Technology given the compound, the descriptors for these compounds found in literature and the descriptors the panellists given on 26.07.2017 (n=15)

Compound	Description literature	Description testers
2,3-Dimethylpyrazine	cocoa, roasted nuts, medical, roasted meat	roasted nuts, intense, slightly green, pyrazine, sweet, peanut butter, like caramel, burnt, fatty, nutty, toasty, malty, slightly fat, slightly like chocolate, hazelnut, bread, wheat, cocoa, raw meat, oily, slightly fishy, phenolic, burnt electronics, burnt rice, like butter, popcorn, like wood, cooked vegetables, smoky
3-Methylbutanol	whiskey, malty, burnt	slightly green, vomit, sweet, slightly like cloves, like metal, overripe banana, fusel alcohol, pungent, slightly like vegetables, slightly fatty, like cheese, intrusive, slightly floral, fruity, apricot, musty, phenolic, concentrated vegetables/herbs, acidic, slightly rancid, nearly like apple cider vinegar, peas, slightly like anise, slightly alcoholic, fresh, ripe fruit, salad marinade
2-ethylfuran	pungent, acidic, sweet, like rubber	alcoholic, green, slightly pungent, like detergent, slightly sweaty, fatty, solvent, medical, musty, woody, acetone, floral, slightly sweet, harsh, slightly oily, salad dressing, old walnut, slightly greenish, toasty, junipers, sticky, coating, glue
Benzaldehyde	almond, burnt sugar	benzaldehyde, almond, marzipan, becomes green, bitter almond, very sweet, intrusive intense, rancid almond, very sweet, cotton candy
Pentanal	almond, malty, pungent	like sweat, acidic, like vomit, sour milk, slightly like hay, nutty, slightly dusty, sweet, green, old grass, like cheese, like milk, slightly rancid, butanoic acid, like sheep cheese, slightly woody, harsh, plant bug, hollow, smoky, fermented
Camphor	camphor	camphor, cooling, green, grassy, woody, sweet, fresh, pungent, slightly like mint, detergent, lime, lemon, leafy green, slightly citrusy, green paprika, pyrazine, forest, eucalyptus, junipers, like menthol
***(E)*-2-Hexenal**	apple, green	green, cut grass, aldehyde, sweet, marzipan, apple, fruity, fatty, slightly like cotton candy, floral, peas, grassy, fruits, apricot, fresh, green trees, rancid, earthy, citrus, green apple, pear, aromatically, restrained

Compound	Description literature	Description testers
(*E, E*) 2,4-Decadienal	fried, fat, wax	plant bug, fatty, rancid, aldehyde, soapy, green, fries, acidic, pungent, green, citrus, orange, slightly smoky, metallic, oily, frying oil, harsh, cooked, slightly like vegetables, slightly like nuts, pungent in the nose
2-ethyl-3,5(6)-Dimethylpyrazine	potato, roasted	intense, nutty, toasty, pungent, slightly green, sweet, hazelnut, dry, spices, solvent, woody, walnut, oily, burnt, burnt electronics, slightly medical, glue, musty, coating, slightly ethereal, wet cloth
2-Acetylpyrolin	nutty, roasted	mushrooms, forest floor, green, fatty, slightly nutty, old frying oil, bitter, green nuts, smoky, fresh, melon, potato, musty, cold smoke, charcoal, cold charcoal, forest, slightly moist, medical, slightly burnt, cellar, earthy, toasty, sweet, paprika
***(E)*-2-Decenal**	orange, greasy	soapy, slightly fat, coriander green, green, fatty, metallic, slightly green, aldehyde, overripe fruits, apple, citrus, floral, musty, sweet, plum, fresh, fruity, more like orange, foamy, cooked, plants, leaves, overripe nuts, rancid, slightly citrusy, green walnut, plant bug, harsh, herbs, ginger
2,3-Butandione	butter	diacetyl, sour butter, slightly like glue, slightly pungent, sweaty, slightly like cheese, like butter, sweet, cream, rancid, butter milk, milk product, rancid yogurt, sour, like milk, solvent, glue, sour cream, curd

Table 27: Description of the substances tested with the sensory panel of Graz University of Technology given the compound, the descriptors for these compounds found in literature and the descriptors the panellists given on 30.08.2017 (n=10)

Compound	Description testers
2-ethylfuran	green, slightly like solvent, slightly floral, slightly sweet, toasty, green notes, slightly sour, nutty, pear, slightly woody, slightly pungent, unripe fruit, wet wood, fresh, slightly harsh, like rubber, peach
Pentanal	like milk, rancid, apple, fatty, oily, sweet, toasty, like vomit, like yeast, bread, flour, raw dough, like cheese, slightly rancid, nutty, almond
(*E*)-2-Decenal	soapy, slightly floral, green, plant bug, fresh, citrus, fatty, like mushrooms, bitter almond, potato, cooked, fatty, rancid
(*E, E*)-2,4-Decadienal	floral, slightly like apple, pungent, green, fruity, slightly citrusy, fresh, peas, rhubarb, fatty, fried, raw chicken, cooked, paprika, slightly like fresh mint
2,3-Dimethylpyrazine	burnt electronics, very dark bread crust, toasty, sweet, popcorn, burnt nuts, slightly rancid, bread crust, cookies, roasted wheat, slightly like caramel, malty, nuts, wheat
2-Ethyl-3,5(6)-Dimethylprazine	pungent, solvent, slightly phenolic, toasty, sweet, nutty, slightly toasted, medical, earthy, malty, coffee beans, roasted almonds, disinfectant
(*E*)-2-Octenal	slightly like cinnamon, slightly like cucumber, plant bug, fresh, slightly citrusy, slightly fatty, slightly pungent, green, like vegetables, woody, cucumber, bergamot

5.2.3.2. Descriptive evaluation

To obtain information on the sensory properties of the types of oil if interest descriptive analysis was performed. Here only still durable oils were tested considering their colour, odour and taste.
For the organic chia seed oil with a BBD of 28.11.2017 most panellists described a green and nutty odour, a yellowish and clear colour and a nutty, slightly fishy and mild taste, the detailed description of all testers can be seen in Table 28. The organic chia seed oil exceeding on 15.05.2018 was found to smell nutty and green, had as a well a yellowish and clear colour and the taste was described as nutty, a bit bitter, scrapie and fishy. All descriptions are gathered in Table 29.

Table 28: Sensory description of the organic chia seed oil exceeding on 28.11.2017, collected descriptors of all panellists (n=15)

Odour	slightly nutty, hay, fresh, green, mild, nutty, slightly toasty, slightly fatty, slightly like vegetables, slightly sweet, spicy, wheat, citrus, slightly flowery, unripe walnuts, green beans, harsh, acidic, furniture polish, tropical timber, green leaves, green hazelnuts, vinegar, fishy, rancid, slightly bitter
Colour	yellowish, clear, light yellow, bright yellow, green reflex, gold-yellow, lemon yellow, amber
Taste	nutty, slightly fishy, malty, slightly like vegetables, mild, unremarkable, sweetish, walnut, black tea, smooth, raspy, slightly like peas, slightly bitter, slightly like wood, harsh, green, slightly astringent, hazelnut, aftertaste like carrots, slightly flaky, creamy texture

Table 29: Sensory description of the organic chia seed oil exceeding on 15.05.2018, collected descriptors of all panellists (n=15)

Odour	nutty, fresh, green, spicy, slightly like vegetables, slightly fatty, slightly sharp, artichokes, candied lemons, hay, dried herbs, weak, wheat, lemon-like, fishy, sparkling sour, dull, musty, nearly rancid, like a cellar, toasty, intense
Colour	yellowish, clear, more intense, bright yellow, lemon yellow, bright borders, cloudy, greenish
Taste	nutty, slightly bitter, slightly raspy, unremarkable, slightly like vegetables, fishy, old, slightly like peas, watery, mild, slightly musty, grassy, green, creamy, toasty, bread crust

The odour of the organic flaxseed oil that exceeded on 05.09.2017 was described as nutty, hay and bread like. The colour was found to be clear and gold-yellow. The taste was said to be bitter and nutty, as seen from Table 30. The flaxseed oil with the BBD on 29.08.2017 was found to be hay-like and green in odour, the colour was again clear and gold-yellow, and the taste was described mostly as bitter. The description of all testers can be seen in
Table 31.

Table 30: Sensory description of the organic flaxseed oil exceeding on 05.09.2017, collected descriptors of all panellists (n=15)

Odour	nutty, white walnut, hay, bread, sour dough, green, yeast, slightly acidic, grassy, vegetables, like wood, oily, slightly rancid, intense, roasted nuts, seeds
Colour	clear, gold yellow, strong yellow, dark yellow, slightly brown, slightly cloudy, egg yolk yellow, faint
Taste	harsh, fresh walnut, bitter, rancid nuts, hay, nutty, like yeast, sharp, bread, malty, fatty, fishy, slightly green, astringent

Table 31: Sensory description of the organic flaxseed oil exceeding on 29.08.2017, collected descriptors of all panellists (n=15)

Odour	nutty, hay, green, sour dough, cooked green beans, slightly pungent, medical, like yeast, seeds, potatoes, rancid, fishy, fatty, fresh, slightly like mustard, herbaceous
Colour	clear, gold yellow, strong yellow, dark yellow, cloudy, very thick
Taste	harsh, like hay, walnut, nutty, bitter, green beans, yeast, nut shells, fruity, woody, slightly sweet, fishy, malty, astringent, green, asparagus, bread, raspy

The organic hempseed oil exceeding on 03.05.2018 was described by the panellists as toasty and nutty in odour as well as in taste and a clear olive-green was described in colour, detailed description can be seen in Table 32. The hempseed oil with a BBD on 03.05.2018 was described in odour as hay-like, toasty, slightly nutty and an odour like rank nuts. The colour was found to be olive-green and the taste was described by most panellists as bitter, grassy, hay-like and nutty, all descriptors named by the sensory test panel can be seen in Table 33.

Table 32: Sensory description of the organic hempseed oil exceeding on 03.05.2017, collected descriptors of all panellists (n=15)

Odour	very nutty, toasty, grassy, nutty, slightly sweet, bread, green, like vegetables, wheat, like hay, slightly like butter, pumpkin seed oil, oily, hazelnuts
Colour	clear, olive green, dark, golden brown, faint
Taste	toasty, slightly bitter, nutty, slightly fat, grassy, hay, walnut, mild, green, slightly harsh, like butter, slightly rancid

Table 33: Sensory description of the hempseed oil exceeding on 03.05.2017, collected descriptors of all panellists (n=15)

Odour	rancid nuts, grassy, hay, toasty notes, slightly nutty, green, slightly pungent, acidic, slightly toasty, fatty, mild, sweet, lipid oxidation, petrol station, lube oil, artificial
Colour	clear, olive green, notes of yellow, brighter, slightly cloudy
Taste	toasty, bitter, grassy, like hay, slightly rancid, nutty, fresh, herbs, burnt, sharp, wheat, old, walnut, green, butter, wood, malty, astringent, mild, rancid, grainy, raspy, pithy

6. Conclusion

The aim of this thesis was to investigate the process induced (oxidative) changes in three highly unsaturated vegetable oils. The three oils were chosen by the oil mill Fandler, as those three oils are on the one hand one of the best-selling oils from the producer and because of their high content of highly unsaturated fatty acids they are very sensible to oxidative changes.

Looking at the fat classification values it was shown that the oxidation parameters, as expected, increased over time. The flaxseed oil only has a shelf life of approximately six months and additionally should be stored in a cool place. This is also visible in the final fat classification numbers and should not be modified in the future. However, the hemp and chia seed oil have a shelf life of one year and can be stored at room temperature, here the fat classification numbers indicate the expected increase over time. The oils are still edible at the end of the best before date. The evaluation of the rapid test method showed that cold pressed highly unsaturated oils are partly not suitable for the determination using this rapid test method. This problem will be tackled by the producer of the device.

The determination of the fatty acid composition showed that the composition is not changing over the course of time and was found to be similar to values found in literature for all three oils. Here the flaxseed and chia seed oil showed a high concentration of approximately 60 % in α-linolenic acid, in contrast to the hempseed oil, which showed a concentration of over 50 % of linoleic acid and a small amount of the rare γ-linolenic acid.

Finally, the overall roasting, pressing and storage process was investigated. As to the best of the knowledge of the author, the roasting, pressing and storage process of these three kinds of oils has not been studied so far. It could be shown that especially in the hempseed oil, the duration and the high temperature of the roasting is needed in order to give the oil its typical roasty and nutty odour and taste. Looking at the chia seed oil, it could be seen that the long storage in the tank had no significant impact on the final quality of the oil. The flaxseed oil is only stored for one day as it would oxidize at a longer storage period and is already settled and clear after one day. Additionally, the oil only has a shelf life of six months. It is one of the best-selling oils at the oil mill Fandler and is therefore pressed every week at the oil mill.

All these findings were supported by the results of the sensory evaluations that were conducted with the sensory expert panel of Graz University of Technology. In addition to the findings during the different determinations of the oils, also new questions arose. More investigations should be carried out into the whole process of the roasting, pressing and storage of the oils. Additional samples of the pressing cake that is left over after the pressing of the oil and the sediment that is formed while the oil is stored in tanks for settling should be gathered.

Besides, the samples from the storage process were taken just one day after the pressing and at the end of the storage period. As these periods can take a few weeks, samples that are taken more often during this period of time could give a better insight into the formation of oxidation products, as well as the development of flavour compounds. To complete these datasets, more sensory evaluations could be performed to see which impact storage has on the changes in the sensory profile of the final product.

Appendix

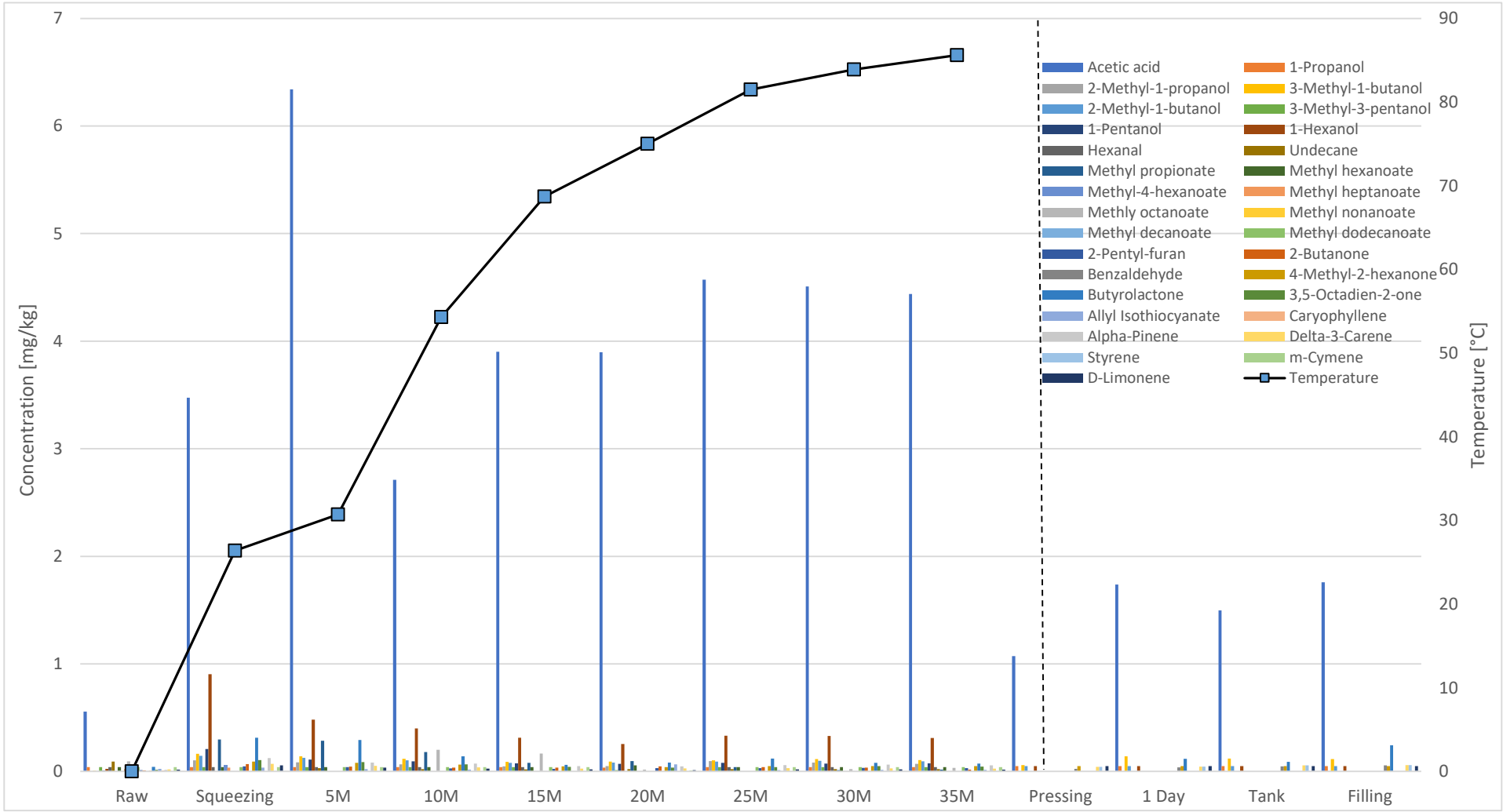


Figure 52: Concentration of all detected compounds found in the roasting, pressing and storage process of chia seeds and chia seed oil given in mg/kg compared to the curve of the roasting temperature, calculated values of the concentration relative to the internal standard of the concentration in mg/kg of measured samples were used (n=4)

Table 34: Calculated values of the concentration relative to the internal standard (2-pentanol) in mg/kg of the measured samples and the calculated OAV (Odour Activity Value) for the compounds an odour threshold value was available given for the raw chia seeds, for the squeezed seeds and for the different roasting steps from 5 to 35 minutes of roasting

Compound	Odour threshold [mg/kg]	Raw	Squeezing	5M	10M	15M	20M	25M	30M	35M
Acetic acid	0.750	0.556	3.475	6.341	2.712	3.902	3.898	4.571	4.509	4.438
OAV		0.742	4.633	8.455	3.616	5.203	5.197	6.094	6.012	5.917
1-Propanol	n.a.	n.d.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
OAV		-	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
2-Methyl-1-propanol	1.000	n.d.	0.105	0.085	0.067	0.048	0.050	0.097	0.084	0.071
OAV		-	0.105	0.085	0.067	0.048	0.050	0.097	0.084	0.071
3-Methyl-1-butanol	0.100	0.006	0.165	0.140	0.118	0.090	0.091	0.103	0.116	0.105
OAV		0.062	1.654	1.402	1.183	0.902	0.909	1.034	1.163	1.053
2-Methyl-1-butanol	0.480	0.004	0.146	0.127	0.105	0.080	0.082	0.091	0.099	0.093
OAV		0.009	0.305	0.265	0.218	0.166	0.170	0.189	0.206	0.195
3-Methyl-3-pentanol	n.a.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
1-Pentanol	0.470	0.007	0.209	0.111	0.094	0.074	0.072	0.080	0.073	0.077
OAV		0.016	0.445	0.237	0.201	0.158	0.152	0.171	0.156	0.163
1-Hexanol	0.400	0.024	0.905	0.483	0.399	0.314	0.256	0.332	0.329	0.312
OAV		0.059	2.263	1.207	0.998	0.784	0.640	0.830	0.822	0.779
Hexanal	0.120	n.q.	n.q.	n.q.	n.d.	n.q.	n.d.	n.q.	n.q.	n.q.
OAV		0.263	0.644	0.039	-	0.022	-	0.026	0.055	0.018
Undecane	5.750	0.092	0.000	0.032	0.023	0.019	0.023	0.022	0.023	0.022
OAV		0.016	0.000	0.006	0.004	0.003	0.004	0.004	0.004	0.004

Methyl propionate	n.a.	0.000	0.298	0.286	0.181	0.079	0.096	0.042	0.013	0.016
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Methyl hexanoate	n.a.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Methyl-4-hexanoate	n.a.	0.000	0.057	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	n.a.	-	-	-	-	-	-	-
Methyl heptanoate	n.a.	0.023	0.036	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	n.a.	-	-	-	-	-	-	-
Methly octanoate	n.a.	0.093	n.d.	n.d.	0.201	0.168	0.018	n.d.	0.022	0.033
OAV		n.a.	-	-.	n.a.	n.a.	n.a.	-.	n.a.	n.a.
Methyl nonanoate	n.a.	0.046	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	-	-	-	-	-	-	-	-
Methyl decanoate	n.a.	0.023	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	-	-	-	-	-	-	-	-
Methyl dodecanoate	n.a.	n.q.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	-	-	-	-	-	-	-	-
2-Pentyl-furan	8.000	0.012	0.047	0.040	0.029	0.025	0.031	0.030	0.033	0.030
OAV		0.001	0.006	0.005	0.004	0.003	0.004	0.004	0.004	0.004
2-Butanone	40.000	0.010	0.069	0.044	0.036	0.036	0.048	0.041	0.037	0.018
OAV		0.000	0.002	0.001	0.001	0.001	0.001	0.001	0.001	n.d.
4-Methyl-2-hexanone	n.a.	0.006	0.093	0.081	0.064	0.047	0.041	0.049	0.050	0-
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Butyrolactone	0.300	0.043	0.313	0.293	0.142	0.063	0.082	0.121	0.081	0.073
OAV		0.142	1.044	0.976	0.473	0.209	0.273	0.402	0.270	0.242

***(E,E)*-3,5-Octadien-2-one**	n.a.	0.015	0.106	0.087	0.067	0.042	0.037	0.040	0.050	0.046
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Allyl Isothiocyanate	n.a.	0.024	0.036	0.023	0.016	0.003	0.066	0.012	0.015	0.014
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Caryophyllene	n.a.	0.006	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	-	-	-	-	-	-	-	-
α-Pinene	0.274	0.016	0.124	0.083	0.074	0.050	0.048	0.059	0.064	0.057
OAV		0.058	0.452	0.303	0.271	0.182	0.175	0.215	0.232	0.209
δ-3-Carene	n.a.	0.020	0.072	0.051	0.037	0.027	0.028	0.030	0.028	0.027
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
m-Cymene	2.510	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
D-Limonene	14.700	0.017	0.057	0.036	0.028	0.019	0.012	0.020	0.019	0.018
OAV		0.001	0.004	0.002	0.002	0.001	0.001	0.001	0.001	0.001

n.a. – OAV Value not available
n.d. – not detectable (LOD < 3)
n.q. – not quantifiable (LOQ < 10)

Table 35: Calculated values of the concentration relative to the internal standard (2-pentanol) in mg/kg of the measured samples and the calculated OAV (Odour Activity Value) for the compounds an odour threshold value was available given for the freshly pressed chia seed oil, for the chia seed oil one day after pressing, for the oil after the sedimentation in the tank and in the filled bottle

Compound	Odour threshold [mg/kg]	Pressing	1 Day after Pressing	Tank	Filling
Acetic acid	0.750	1.071	1.736	1.496	1.759
OAV		1.429	2.315	1.995	2.346
3-Methyl-1-butanol	0.100	0.058	0.141	0.121	0.114
OAV		0.584	1.408	1.211	1.143
2-Methyl-1-butanol	0.480	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.
1-Hexanol	0.400	0.150	0.291	0.295	0.410
OAV		0.374	0.728	0.737	1.026
Benzaldehyde	0.060	0.023	0.039	0.049	0.057
OAV		0.385	0.644	0.809	0.949
4-Methyl-2-hexanone	n.a.	n.q.	n.q.	n.q.	n.q.
OAV		n.a.	n.a.	n.a.	n.a.
Butyrolactone	0.300	0.000	0.118	0.090	0.244
OAV		0.000	0.394	0.302	0.814
Styrene	7.650	0.043	0.046	0.057	0.059
OAV		0.006	0.006	0.007	0.008
m-Cymene	2.510	n.d.	n.d.	n.d.	n.d.
OAV		n.d.	n.d.	n.d.	n.d.
D-Limonene	14.700	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.

n.a. – OAV Value not available

n.d. – not detectable (LOD < 3)

n.q. – not quantifiable (LOQ < 10)

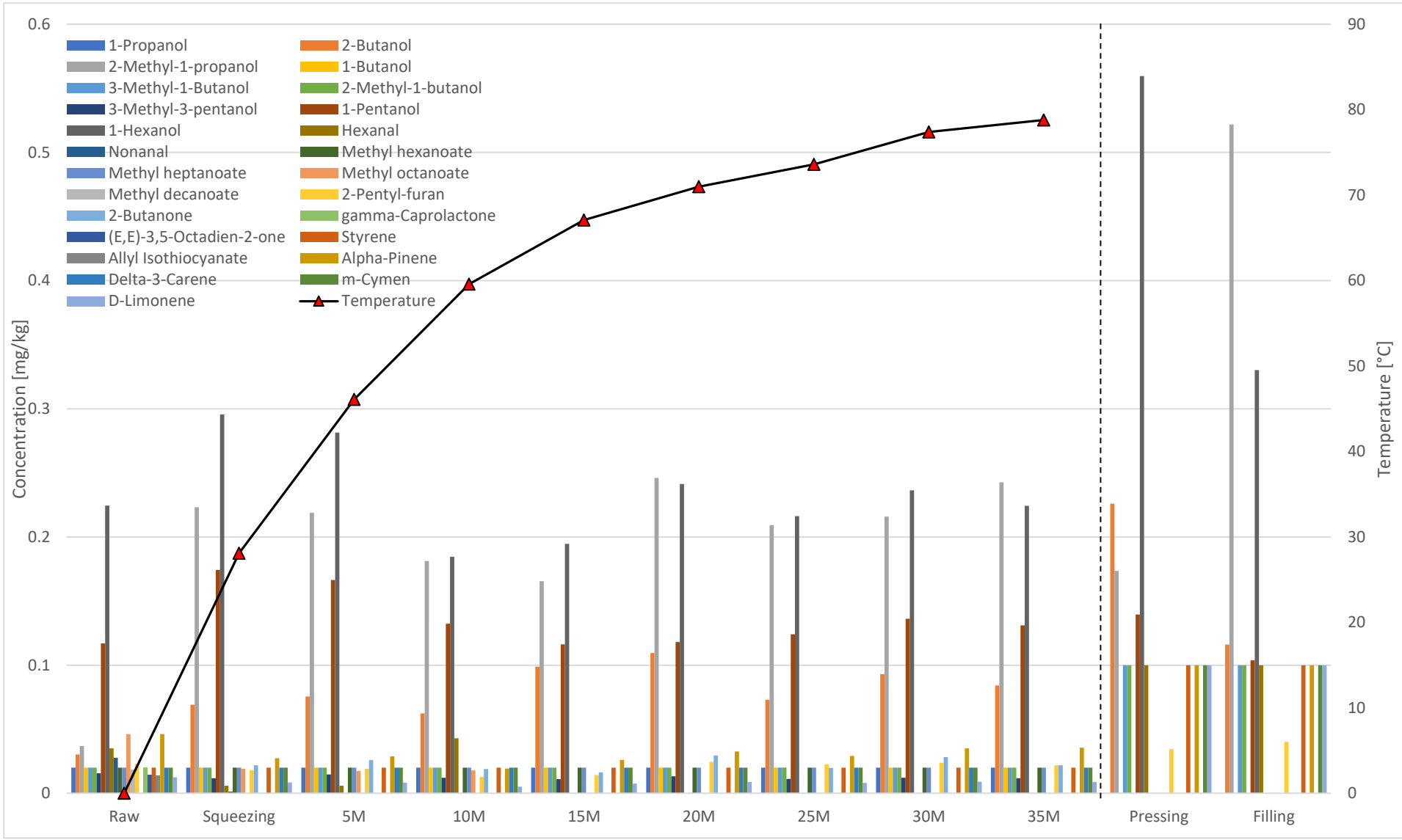


Figure 53: Concentration of all detected compounds found in the roasting, pressing and storage process of flaxseeds and flaxseed oil given in mg/kg compared to the curve of the roasting temperature, calculated values of the concentration relative to the internal standard of the concentration in mg/kg of measured samples were used (n=4)

Table 36: Calculated values of the concentration relative to the internal standard (2-pentanol) in mg/kg of the measured samples and the calculated OAV (Odour Activity Value) for the compounds an odour threshold value was available given for the raw flaxseeds, the squeezed seeds, the roasted seeds between 5 and 35 minutes

Compound	Odour threshold [mg/kg]	Raw	Squeezing	5M	10M	15M	20M	25M	30M	35M
1-Propanol	n.a.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
2-Butanol	0.500	0.030	0.069	0.076	0.062	0.099	0.110	0.073	0.093	0.084
OAV		0.061	0.138	0.151	0.125	0.198	0.219	0.146	0.186	0.168
2-Methyl-1-propanol	1.000	0.037	0.223	0.219	0.181	0.165	0.246	0.209	0.216	0.243
OAV		0.037	0.223	0.219	0.181	0.165	0.246	0.209	0.216	0.243
1-Butanol	0.038	0.000	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
OAV		0.000	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3-Methyl-1-Butanol	0.100	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
2-Methyl-1-butanol	0.480	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3-Methyl-3-pentanol	n.a.	0.016	0.012	0.015	0.012	0.011	0.013	0.011	0.012	0.012
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
1-Pentanol	0.470	0.117	0.174	0.166	0.132	0.116	0.118	0.124	0.136	0.131
OAV		0.249	0.371	0.354	0.282	0.247	0.251	0.264	0.290	0.279
1-Hexanol	0.400	0.224	0.296	0.282	0.185	0.195	0.241	0.216	0.236	0.224
OAV		0.561	0.739	0.704	0.461	0.487	0.603	0.541	0.591	0.561
Hexanal	0.120	0.035	0.006	0.006	0.043	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		0.292	0.049	0.049	0.358	-	-	-	-	-
Nonanal	1.000	0.028	0.001	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		0.028	0.001	-	-	-	-	-	-	-
Methyl hexanoate	n.a.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.

OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Methyl heptanoate	n.a.	n.q.	n.q.	n.q.	n.q.	0.000	0.000	n.q.	n.q.	n.q.
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Methyl octanoate	n.a.	0.046	0.019	0.017	0.018	0.000	0.000	0.000	0.000	0.000
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Methyl decanoate	n.a.	0.019	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	-	-	-	-	-	-	-	-
2-Pentyl-furan	8.000	0.023	0.018	0.019	0.013	0.014	0.025	0.023	0.024	0.022
OAV		0.003	0.002	0.002	0.002	0.002	0.003	0.003	0.003	0.003
2-Butanone	40.000	0.000	0.022	0.026	0.019	0.016	0.030	0.020	0.028	0.022
OAV		0.000	0.001	0.001	n.d.	n.d.	0.001	0.000	0.001	0.001
Styrene	7.650	n.q.	n.q.	n.q.	-.	-	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
Allyl Isothiocyanate	n.a.	0.014	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		0.000	-	-	-	-	-	-	-	-
α-Pinene	0.274	0.046	0.027	0.029	0.019	0.026	0.033	0.029	0.035	0.036
OAV		0.169	0.100	0.105	0.071	0.095	0.120	0.107	0.128	0.130
δ-3-Carene	n.a.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
m-Cymene	2.510	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.d.	n.d.
OAV		n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	-	-
D-Limonene	14.700	0.013	0.008	0.008	0.005	0.008	0.009	0.008	0.009	0.009
OAV		0.001	0.001	0.001	0.000	0.001	0.001	0.001	0.001	0.001

n.a. – OAV Value not available
n.d. – not detectable (LOD < 3)
n.q. – not quantifiable (LOQ < 10)

Table 37: Calculated values of the concentration relative to the internal standard (2-pentanol) in mg/kg of the measured samples and the calculated OAV (Odour Activity Value) for the compounds an odour threshold value was available given for the freshly pressed flaxseed oil and as the flaxseed oil is filled one day after pressing the value for filling

Compound	Odour threshold [mg/kg]	Pressing	Filling
2-Butanol	0.500	0.226	0.116
OAV		0.452	0.232
2-Methyl-1-propanol	1.000	0.174	0.522
OAV		0.174	0.522
3-Methyl-1-Butanol	0.100	n.q.	n.q.
OAV		n.q.	n.q.
2-Methyl-1-butanol	0.480	n.q.	n.q.
OAV		n.q.	n.q.
1-Pentanol	0.470	0.140	0.104
OAV		0.297	0.221
1-Hexanol	0.400	0.560	0.330
OAV		1.399	0.826
Hexanal	0.120	n.q.	n.q.
OAV		n.q.	n.q.
2-Pentyl-furan	8.000	0.034	0.040
OAV		0.004	0.005
Styrene	7.650	n.q.	n.q.
OAV		n.q.	n.q.
α-Pinene	0.274	n.q.	n.q.
OAV		n.q.	n.q.
m-Cymene	2.510	n.q.	n.q.
OAV		n.q.	n.q.
D-Limonene	14.700	n.q.	n.q.
OAV		n.q.	n.q.

n.a. – OAV Value not available
n.d. – not detectable (LOD < 3)
n.q. – not quantifiable (LOQ < 10)

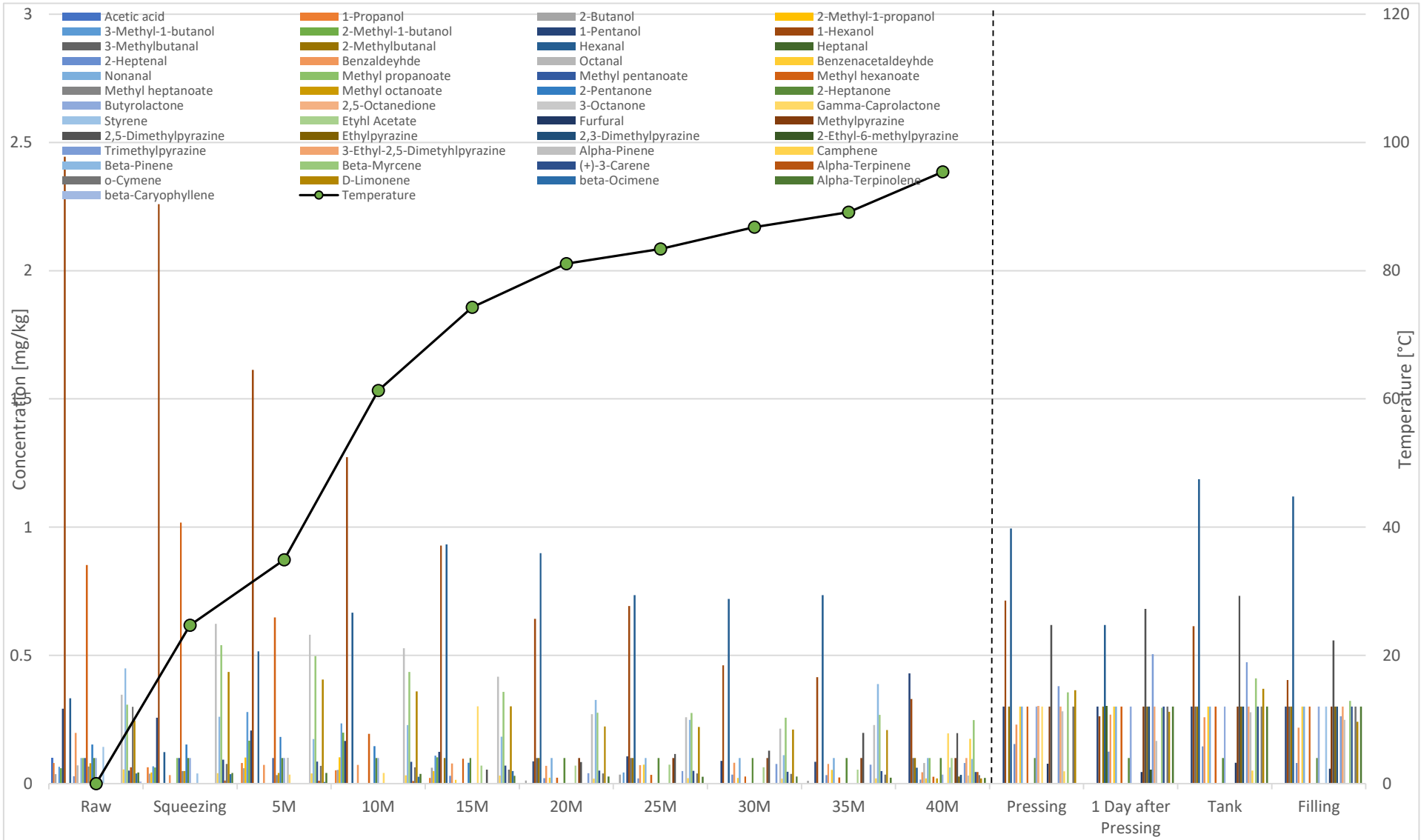


Figure 54: Concentration of all detected compounds found in the roasting, pressing and storage process of hempseeds and hempseed oil given in mg/kg compared to the curve of the roasting temperature, calculated values of the concentration relative to the internal standard of the concentration in mg/kg of measured samples were used (n=4)

Table 38: Calculated values of the concentration relative to the internal standard (2-pentanol) in mg/kg of the measured samples and the calculated OAV (Odour Activity Value) for the compounds an odour threshold value was available given for the raw hempseeds, for the squeezed seeds and for the different roasting steps from 5 to 40 minutes of roasting

Compound	Odour threshold [mg/kg]	Raw	Squeezing	5M	10M	15M	20M	25M	30M	35M	40M
Acetic acid	0.750	n.q.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.q.	-	-	-	-	-	-	-	-	-
1-Propanol	n.a.	0.080	0.064	0.080	0.052	0.022	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	-	-	-	-	-
2-Butanol	0.500	0.037	0.039	0.060	0.054	0.061	0.012	0.035	n.d.	0.011	n.d.
OAV		0.074	0.077	0.121	0.108	0.123	0.023	0.070	-	0.021	-
2-Methyl-1-propanol	1.000	0.000	0.044	0.101	0.102	0.049	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		0.000	0.044	0.101	0.102	0.049	-	-	-	-	-
3-Methyl-1-butanol	0.100	0.066	0.068	0.279	0.235	0.109	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		0.658	0.676	2.793	2.350	1.086	-	0.433	-	-	-
2-Methyl-1-butanol	0.480	0.060	0.063	0.167	0.199	0.102	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		0.125	0.130	0.347	0.414	0.213	-	-	-	-	-
1-Pentanol	0.470	0.292	0.257	0.207	0.166	0.124	0.087	0.106	0.089	0.085	0.430
OAV		0.621	0.547	0.440	0.354	0.263	0.185	0.225	0.189	0.180	0.915
1-Hexanol	0.400	2.445	2.259	1.613	1.273	0.928	0.642	0.692	0.462	0.415	0.330
OAV		6.112	5.649	4.032	3.182	2.320	1.606	1.731	1.154	1.037	0.825
3-Methylbutanal	0.005	n.d.	n.d.	n.d.	n.d.	n.d.	n.q.	n.q.	n.d.	n.d.	n.q.
OAV		-	-	-	-	-	n.q.	n.q.	-	-	-
2-Methylbutanal	0.005	n.d.	n.d.	n.d.	n.d.	n.q.	n.q.	n.q.	n.d.	n.d.	n.q.
OAV		-	-	-	-	n.q.	n.q.	n.q.	-	-	n.q.
Hexanal	0.120	0.333	0.122	0.516	0.667	0.932	0.898	0.735	0.720	0.735	0.062
OAV		2.772	1.020	4.300	5.555	7.769	7.486	6.123	5.997	6.126	0.513
2-Heptenal	1.500	0.028	n.d.	n.d.	n.d.	0.030	0.021	0.020	0.035	0.033	0.015
OAV		0.019	-	-	-	0.020	0.014	0.014	0.023	0.022	0.010
Benzaldeyhde	0.060	0.197	0.033	0.072	0.073	0.078	0.069	0.072	0.082	0.077	0.045
OAV		3.288	0.550	1.208	1.212	1.306	1.146	1.205	1.361	1.281	0.745
Octanal	0.320	0.071	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.080
OAV		0.222	-	-	-	-	-	-	-	-	0.250

Benzenacetaldeyhde	0.040	n.d.	n.d.	n.d.	0.001	0.014	0.023	0.074	0.022	0.053	0.020
OAV		-	-	-	0.027	0.355	0.580	1.849	0.561	1.327	0.498
Nonanal	1.000	n.q.	n.d.	n.d.	n.d.	n.d.	n.q.	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	-	-	-	-	n.q.	n.q.	n.q.	n.q.	n.q.
Methyl propanoate	n.a.	n.q.	n.q.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.075
OAV		n.a.	n.a.	-	-	-	-	-	-	-	n.a.
Methyl pentanoate	n.a.	n.q.	n.q.	n.q.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	n.a.	n.a.	-	-	-	-	-	-	-
Methyl hexanoate	n.a.	0.852	1.017	0.648	0.194	0.097	0.023	0.034	0.028	0.024	0.027
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Methyl heptanoate	n.a.	0.067	0.049	0.033	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	n.a.	n.a.	-	-	-	-	-	-	-
Methyl octanoate	n.a.	0.079	0.049	0.041	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.020
OAV		n.a.	n.a.	n.a.	-	-	-	-	-	-	n.a.
2-Pentanone	n.a.	n.q.	n.q.	n.q.	n.q.	n.q.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	-	-	-	-	-
2-Heptanone	0.300	n.q.	n.q.	0.092	0.090	0.078	0.056	0.058	0.059	0.059	0.062
OAV		n.q.	n.q.	0.306	0.300	0.258	0.187	0.193	0.195	0.198	0.208
Butyrolactone	n.a.	n.q.	n.q.	n.q.	n.q.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	n.a.	n.a.	n.a.	-	-	-	-	-	-
3-Octanone	n.a.	n.d.	n.d.	n.q.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		-	-	n.a.	n.a.	-	-	-	-	-	-
γ-Caprolactone	n.a.	n.d.	n.d.	0.035	0.042	0.302	n.d.	n.d.	n.d.	n.d.	0.196
OAV		-	-	n.a.	n.a.	n.a.	-	-	-	-	n.a.
Styrene	7.650	0.144	0.040	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.062
OAV		0.019	0.005	-	-	-	-	-	-	-	0.008
Etyhl Acetate	7.650	n.d.	n.d.	n.d.	n.d.	0.070	0.070	0.074	0.063	0.054	n.q.
OAV		-	-	-	-	0.009	0.009	0.010	0.008	0.007	n.q.
Methylpyrazine	27.000	n.d.	n.d.	n.d.	n.d.	n.d.	n.q.	n.q.	n.q.	n.q.	n.q.
OAV		-	-	-	-	-	n.q.	n.q.	n.q.	n.q.	n.q.
2,5-Dimethylpyrazine	2.600	n.d.	n.d.	n.d.	n.d.	0.055	0.084	0.115	0.128	0.198	0.088
OAV		-	-	-	-	0.021	0.032	0.044	0.049	0.076	0.034
Ethylpyrazine	17.000	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.069
OAV		-	-	-	-	-	-	-	-	-	0.004

2,3-Dimethylpyrazine	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.160
OAV		-	-	-	-	-	-	-	-	-	n.a.
Trimethylpyrazine	0.270	n.d.	n.d.	n.d.	n.d.	n.d.	0.041	0.049	0.077	0.074	0.080
OAV		-	-	-	-	-	0.151	0.182	0.284	0.273	0.297
3-Ethyl-2,5-Dimetyhlpyrazine	0.024	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.q.
OAV		-	-	-	-	-	-	-	-	n.d.	n.q.
α-Pinene	0.274	0.346	0.623	0.580	0.528	0.417	0.271	0.258	0.215	0.228	0.031
OAV		1.264	2.275	2.118	1.927	1.521	0.989	0.943	0.784	0.833	0.114
Camphene	n.a.	0.055	0.040	0.040	0.033	0.031	0.019	0.022	0.019	0.020	0.175
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
β-Pinene	430.000	0.449	0.260	0.174	0.228	0.183	0.326	0.249	0.111	0.388	0.096
OAV		0.001	0.001	0.000	0.001	0.000	0.001	0.001	0.000	0.001	0.000
β-Myrcene	0.790	0.308	0.540	0.497	0.435	0.358	0.276	0.275	0.256	0.268	0.248
OAV		0.390	0.683	0.629	0.551	0.453	0.349	0.348	0.325	0.339	0.314
δ-3-Carene	n.a.	0.051	0.093	0.086	0.085	0.071	0.051	0.049	0.046	0.049	0.045
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
α-Terpinene	n.a.	0.064	0.012	0.011	0.010	n.d.	n.d.	n.d.	n.d.	n.d.	0.045
OAV		n.a.	n.a.	n.a.	n.a.	-	-	-	-	-	n.a.
m-Cymene	2.510	0.299	0.077	0.069	0.064	0.054	0.039	0.040	0.037	0.035	0.033
OAV		0.119	0.031	0.027	0.025	0.022	0.016	0.016	0.015	0.014	0.013
D-Limonene	14.700	0.245	0.435	0.406	0.359	0.302	0.223	0.221	0.211	0.209	0.020
OAV		0.017	0.030	0.028	0.024	0.021	0.015	0.015	0.014	0.014	0.001
β-Ocimene	n.a.	0.040	0.037	0.006	0.026	0.049	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
α-Terpinolene	n.a.	0.043	0.042	0.041	0.038	0.031	0.027	0.027	0.028	0.023	0.022
OAV		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
β-Caryophyllene	n.a.	0.008	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OAV		n.a.	-	-	-	-	-	-	-	-	-

n.a. – OAV Value not available
n.d. – not detectable (LOD < 3)
n.q. – not quantifiable (LOQ < 10)

Table 39: Calculated values of the concentration relative to the internal standard (2-pentanol) in mg/kg of the measured samples and the calculated OAV (Odour Activity Value) for the compounds an odour threshold value was available given for the freshly pressed hempseed oil, for the hempseed oil one day after pressing, for the oil after the sedimentation in the tank and in the filled bottle

Compound	Odour threshold [mg/kg]	Pressing	1 Day after Pressing	Tank	Filling
1-Pentanol	0.470	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.
1-Hexanol	0.400	0.713	0.262	0.614	0.404
OAV		1.783	0.655	1.534	1.010
3-Methylbutanal	0.005	n.d.	n.d.	n.q.	n.q.
OAV		-	-	n.q.	n.q.
2-Methylbutanal	0.005	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.
Hexanal	0.120	0.995	0.618	1.187	1.119
OAV		8.289	5.151	9.892	9.325
Heptanal	0.250	n.d.	n.q.	n.d.	n.d.
OAV		-	n.q.	-	-
2-Heptenal	1.500	0.154	0.125	0.145	0.080
OAV		0.103	0.083	0.097	0.053
Benzaldeyhde	0.060	0.230	0.269	0.258	0.218
OAV		3.841	4.481	4.307	3.629
Benzenacetaldeyhde	0.040	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.
Nonanal	1.000	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.
Methyl hexanoate	n.a.	n.q.	n.q.	n.q.	n.q.
OAV		n.a.	n.a.	n.a.	n.a.
2-Heptanone	0.300	0.103	0.117	0.147	0.101
OAV		0.000	0.000	0.000	0.000
Butyrolactone	n.a.	0.261	0.147	0.297	0.117
OAV		n.a.	n.a.	n.a.	n.a.
2,5-Octanedione	n.a.	n.q.	n.d.	n.d.	n.d.
OAV		n.a.	-	-	-
γ-Caprolactone	n.a.	n.q.	n.d.	n.d.	n.d.
OAV		n.a.	-	-	-
Styrene	7.650	n.d.	n.d.	n.d.	n.q.
OAV		-	-	-	n.q.
Furfural	n.a.	0.078	0.045	0.081	0.058
OAV		n.a.	n.a.	n.a.	n.a.
Methylpyrazine	27.000	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.
2,5-Dimethylpyrazine	2.600	0.618	0.681	0.732	0.559

OAV		0.238	0.262	0.282	0.215
Ethylpyrazine	17.000	n.d.	n.q.	n.q.	n.q.
OAV		-	n.q.	n.q.	n.q.
2,3-Dimethylpyrazine	n.a.	n.d.	n.q.	n.q.	n.q.
OAV		-	n.a.	n.a.	n.a.
2-Ethyl-6-methylpyrazine	n.a.	n.d.	0.055	n.d.	n.d.
OAV		n.a.	n.a.	n.a.	n.a.
Trimethylpyrazine	0.270	0.379	0.505	0.473	0.262
OAV		1.405	1.869	1.753	0.972
3-Ethyl-2,5-Dimetyhlpyrazine	0.024	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.
α-Pinene	0.274	0.282	0.166	0.278	0.249
OAV		1.029	0.606	1.016	0.909
Camphene	n.a.	n.q.	n.d.	n.q.	n.d.
OAV		n.a.	n.a.	n.a.	n.a.
β-Myrcene	0.790	0.356	0.296	0.411	0.323
OAV		0.450	0.375	0.520	0.409
δ-3-Carene	n.a.	n.d.	n.q.	n.q.	n.q.
OAV		n.a.	n.a.	n.a.	n.a.
m-Cymene	2.510	n.q.	n.q.	n.q.	n.q.
OAV		n.q.	n.q.	n.q.	n.q.
D-Limonene	14.700	0.364	0.280	0.370	0.241
OAV		0.025	0.019	0.025	0.016
α-Terpinolene	n.a.	n.d.	n.q.	n.q.	n.q.
OAV		n.a.	n.a.	n.a.	n.a.

n.a. – OAV Value not available

n.d. – not detectable (LOD < 3)

n.q. – not quantifiable (LOQ < 10)

Deskriptive Beurteilung von Chia-Ölen

Name:	
Prüfer Nr.:	

Prüfanleitung: Du bekommst 2 Chia-Ölproben. Versuche bitte, die Öle hinsichtlich aller sensorischer Eigenschaften so gut wie möglich zu beschreiben! Die unten aufgelisteten Attribute können müssen aber nicht verwendet werden.

Beschriebene sensorische Eindrücke:

Geruch: leicht nussig, mild, frische grüne Blätter, nach weißen Bohnen, nach Artischocke
Farbe: klar, gelblich, grüne Reflexe, leuchtend, zitronengelb
Geschmack: arteigener Geschmack, mild nussig, leichte Gemüsenote, leicht bitter, fischig, leicht kratzend

Probennr.	**Geruch**	**Farbe**	**Geschmack**
265 Bio Chiaöl			
411 Bio Chiaöl			

Deskriptive Beurteilung von Lein-Ölen

Name:	
Prüfer Nr.:	

Prüfanleitung: Du bekommst 2 Lein-Ölproben. Versuche bitte, die Öle hinsichtlich aller sensorischer Eigenschaften so gut wie möglich zu beschreiben! Die unten aufgelisteten Attribute können müssen aber nicht verwendet werden.

Beschriebene sensorische Eindrücke:

Geruch: nussig, saatig, heuig, hefig nach Brot, nach frisch geriebenen Walnüssen
Farbe: klar, gelb, strahlend, sonnengelb, hell gelb - grün bis goldgelb
Geschmack: nussig, leicht bitter, herb, heuig, saatig, leicht süß, brotig, Walnüsse, malzig

Probennr.	Geruch	Farbe	Geschmack
987 Bio Leinöl			
312 Leinöl			

Deskriptive Beurteilung von Hanf-Ölen

Name:	
Prüfer Nr.:	

Prüfanleitung: Du bekommst 2 Hanf-Ölproben. Versuche bitte, die Öle hinsichtlich aller sensorischer Eigenschaften so gut wie möglich zu beschreiben! Die unten aufgelisteten Attribute können müssen aber nicht verwendet werden.

Beschriebene sensorische Eindrücke:

Geruch: dezent nussig, heuig, grasig, röstig, säuerlich, nach brauner Butter
Farbe: **Ungeröstet:** grüngelb, tiefgrün **Geröstet:** braun-grün, olivgrün, gelber Schimmer
Geschmack: röstig, nussig, leicht bitter, kernig, heuig, grasig, Sauerampfer, Avocadobutter

Probennr.	**Geruch**	**Farbe**	**Geschmack**
176 Bio Hanföl			
654 Hanföl			

Geruchsbeschreibung

Name	
Prüfer-Nr.	

Prüfanleitung: 1. Auf dem Prüfplatz befinden sich Riechstreifen in Zellophanhüllen. Die Prüfproben sind in der angegebenen Reihenfolge zu prüfen.
2. Der Geruch der Proben ist so genau wie möglich zu beschreiben.

Nr.	Geruchsbeschreibung
1	**2,3-Dimethylpyrazin (G66)** **Kakao, geröstete Nuss, gebratenes Fleisch, medizinisch**
2	**3-Methylbutanol (G197)** **Whiskey, malzig, verbrannt**
3	**2-Ethylfuran (G148)** **gummiartig, stechend, sauer, süß**
4	**Benzaldehyd (G2)** **Mandel, verbrannter Zucker**
5	**Pentanal (G146)** **Mandel, malzig, stechend**
6	**Campher (G 6)** **Kampfer**
7	**2-Hexenal (G17)** **Apfel, grün**
8	**Decadienal (G209)** **frittiert, wachsig, fett**
9	**2-ethyl-3,5(6)-Dimethylpyrazin (G200)** **Kartoffel, gebraten**
10	**2-Acetylpyrolin (G199)** **nussig, gebraten**
11	**2-Decenal (G89)** **schmierig, Orange**

12	**2,3-Butandion (G8)** **Butter, Sauerrahm**

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