CHEMICAL CHARACTERIZATION OF CAMELINA SEED OIL

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ABSTRACT OF THE THESIS

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Camelina sativa (L).Crantz also known as false flax, Dutch flax is an ancient oil seed crop that belongs to the Brassicaceae family. Camelina oil pressed from the seeds of this crop has a unique aroma.

Eighteen camelina oil samples were analyzed for fatty acid composition (13 unrefined, 2 deodorized and 3 refined samples). Eight of these samples were analyzed for unsaponifiables content, free fatty acids and volatiles and semi-volatile compounds. Seven camelina seed samples were analyzed for volatile and semi-volatile compounds as well to determine the suitability of these products in animal feed formulations.

Fatty acid composition was obtained by the trans-esterification of the triacylglycerols in the oil to their methyl esters and 21 different fatty acids with chain length from C-14 to C-24 were identified. The major fatty acids were α-linolenic, linoleic, oleic, eicosenoic and palmitic acid and three fatty acids, namely tricosanoic, pentadecanoic and heptadecanoic are being first reported here.

The unsaponifiables fraction in camelina oil samples ranged between 0.45-0.8% and 21 compounds were identified. The major compounds identified were β-sitosterol, campesterol, cholesterol, phytol, squalene and brassicasterol which accounted for 80-90% of the unsaponifiable content in camelina oil.

A total of 168 and 306 volatile and semi-volatile compounds were identified in the headspace of camelina seeds and oil respectively. Homologous series of lipid oxidation derived compounds like aldehydes, ketones, alcohols, furans and hydrocarbons dominate the aroma and favor profile of the oil and seeds. Sulfur compounds (methyl mercaptan, dimethyl sulfide, dimethyl disulfide and dimethyl trisulfide), naturally occurring 3-alkyl-2-methoxy pyrazines, terpenes, short chain free fatty acids and maillard reaction products were also identified in camelina seeds. The presence of 2-sec-butyl-3-methoxy pyrazine, aldehydes and alcohols (with green notes) and sulfur compounds like 2, 4, 5-trithiahexane and 1-butene-4-isothiocyanato in some camelina oils, may be responsible for the unique aroma of this oil.

The information from this study may potentially be used by camelina oil producers as supporting data for the chemical characteristics of the oil produced in Montana, USA. Camelina oil can serve as a good vegetable source of α-linolenic acid provided it gets the much awaited GRAS certification.

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1.1 Background on *Camelina sativa*

Camelina Crop

Camelina sativa, (L.) Crantz is an ancient oilseed crop that belongs to the family Cruciferae (Brassicaceae) and some examples of the Brassicaceae family include oilseeds like mustard, rapes, canola, crambe and vegetables like cabbage, cauliflower and broccoli. Camelina is also known as false flax, Dutch flax, German sesame, Siberian oilseed and Gold of Pleasure. Camelina crop originated in Germany about 600 B.C. and later spread to Central Europe (Budin *et. al*., 1995). Archeological excavations in Europe and Scandinavia have revealed the existence of *Camelina sativa* (L.) Crantz, C. *microcarpa* and *C.linicola* in the Bronze Age (1500-400 B.C.) and Iron Age (400 B.C.-500 A.D.). It was believed that the seeds of *C.linicola* were consumed along with flaxseeds and cereals primarily in the form of porridge and bread (Zubr, 1997).

Camelina sativa is an annual summer or wintering plant which has branched smooth or hairy stems that become woody at maturity and reach heights ranging from 1-3 feet (Fig.1). The leaves are arrow-shaped, 5-8 cm long with smooth edges and each stem bears small yellow flowers that are pale yellow to green with 4 petals (5-7 mm in diameter).

Camelina is a short-seasoned (85-100 days) crop, and can be grown under different climatic and soil conditions with the exception of heavy clay and organic soil (Zubr, 1997). Camelina is a low-input crop with minimum nutrient requirements and can grow well in low-fertility or saline soils when compared to other oilseed crops like canola, soybean or sunflower.

Figure 1: Camelina Plant

(*Camelina sativa*, (L.)Crantz)

(http://en.wikipedia.org/wiki/File:Camelina_sativa_eF.jpg)

Chemical plant protection is generally not required and camelina is fairly tolerant to insects and weeds. Camelina can survive in extremely low temperature conditions and is well adapted to the colder climatic conditions that prevail in the northerly regions of North America, Europe and Asia (Budin et al., 1995).

Camelina Seed

Pear-shaped capsules (usually 5 mm in diameter) contain close to 10-25 oval shaped Camelina seeds which are yellow in color. The color of the seeds turns dark-brown or reddish on ripening and under storage. Camelina seeds are extremely small that close to 1000 seeds weighs 1.0 g depending on the variety and growth conditions (A. Schuster and W.Friedt, 1998). Figures 2 and 3 are pictures showing the small size of camelina seeds. Camelina crop can be harvested using a combine harvester but care must be taken to avoid the shattering of the seeds during harvesting. Humid and unfavorable weather conditions can lead to seed damage and lower seed yield (Zubr, 1996).

Moisture content in the seeds at the time of harvesting is approximately 11.0 % and is reduced to less than 8 % for safe storage. Camelina seeds contain an oil content of 30-40% DM, crude protein 45 % DM and crude fiber 12.5-17 % fat free DM . Camelina seeds taste bitter with a cabbage trunk or kohlrabi type flavor (Mikersch, 1952). Camelina seeds also contain various aromatic and glucosidic compounds. Glucosinolates are a group of organic compounds that contain sulfur and nitrogen and are derived from glucose and an amino acid. Total glucosinolate content of 13.2 to 36.2 umol/g dry seeds was reported and the level of glucosinolate is dependent on the environment and available sulfur from a particular location (A. Schuster and Friedt, 1998). The β-Glucan

Figure 2: Camelina Capsule and seeds inside the Capsule

(http://seattletimes.nwsource.com/ABPub/2007/11/20/2004026049.jpg)

Figure 3: Camelina Seeds

http://www.canpressco.com/images/camelinaSeedSmall.JPG

content of camelina seeds was 0.4% and this was comparable to other oilseed crops (Budin et al., 1995).

Camelina Oil

Camelina oil is the main product from camelina seeds and the average yield of oil from the seeds is 30-40%DM (Budin et al., 1995, Rode, 2002, Zubr, 2003). It is a golden yellow color liquid with a mild nutty and characteristic mustard aroma. Some of the physical properties of camelina oil reported are refractive index 1.4756 (at 25° C), density 0.92 g/cc(at 25 ° C),iodine number 105 (g I_2/100 g oil) and saponification value 187.8 (mg KOH/g oil) (H.Abramovic and V.Abram , 2005). Carotenoids and chlorophyll have been reported in camelina oil using UV spectroscopy (Sizova et al., 2003).

Camelina Oil-cakes

Extraction of oil from oilseeds by pressing yields a by-product called Oil cakes/ meal which contain residual oil, crude protein and fiber. Camelina oilcakes/meal consists of 5-10 % residual oil, 45 % crude protein, 13 % fiber, 5 % of minerals and some minor levels of vitamins (Zubr, 1997). Mikersch (1952) reported the residual oil content as 13 %, ash (6.6%), crude fiber (11.7%), protein (32.8%) and non-nitrogenous matter (27.2%) in camelina press cake. Because of the high crude protein content, oilcakes are considered economically important and can be used as nutritive supplement in animal feed formulations. A study on crude protein (42.5 %) and amino acids in camellia seeds revealed the presence of 18 amino acids, of which 9 amino acids are essential (Zubr, 2003). Essential amino acids identified were arginine, glycine, isoleucine, leucine, lysine, phenylalanine, proline, threonine, valine, methionine and cystine. The amino acid profile of *Camelina sativa* was comparable to that of other oilseeds like rapeseed, soybean and

flax. The protein content of camelina is excellent for use in animal feed supplements, but Trypsin Inhibitor Activity (TIA, 12-28 mg/g) and presence of glucosinolates could be limiting factors. Budin et al., 1995 had suggested that plant breeding programs could reduce the TIA in Camelina and it should be noted that TIA was reduced in soybean meal from 22.1 to 2.1 mg/g by heating for 40 minutes (Zubr, 2002). A similar cooking process like heating, baking, etc could help in reducing the TIA in camelina seeds and meal to very low levels.

Matthaus and Zubr (2000) reported the presence of 3 glucosinolates in camelina meal and Glucocamelinin (10-methylsulfinyldecyl-Gls) accounted for 62-72% of the total. The other two were 9-methylsulfinylnonyl-Gls (9-MSG) and 11-methyl sulfinyl undecyl-Gls (11-MSG). The total glucosinolates amounted to 14.5 to 24 umol /g. These levels in camelina are considered as moderate to low when compared to other cruciferous oilseeds like crambe (100 umol/g), mustard (120 umol/g) and rape (10-20 umol/g). The glucosinolates in camelina contain long side-chains and hence the enzymatic hydrolysis of these resulted in only non-volatile isothiocyanates and since camelina seeds do not contain progroitrin there is no formation of toxic goitrin (the aglucone of glucosinolates in camelina does not contain an OH group). It can be assumed that the glucosinolates in camelina do not form compounds like sinigrin and progoitrin and camelina meal can be used as a nutritive supplement in animal feedstuff (Matthaus and Zubr, 2000).

Sinapine (which is responsible for the bitter taste in certain vegetables like brussel sprouts,(Belitz and Grosch,1999)) has been reported in Camelina meal (1.7 to 4.2 mg/g) but this value is much lesser than those found in other Brassicaceae oilseeds such as rapeseed (7 mg/g) and mustard (13 mg/g). Total inositol phosphates (21.9-30.1 mg/g),

condensed tannins (1.0-2.4 mg/g expressed as catechin) and heavy metals such as Cadmium (125.4 µg/kg), Nickel (2.4 mg/Kg) and Zinc (49.9 mg/Kg) have been reported in camelina meal (Matthaus and Zubr, 2000). The levels of heavy metals in camelina seeds is much lower than those values reported in camelina meal and it is assumed that the heavy metals are not passed into the oil during oil extraction. Cadmium is considered non-essential and toxic however Nickel (0.4 mg/day) and Zinc (6-22 mg/day) are essential for the functioning of various enzymes and proteins (Belitz and Grosch, 1999). Condensed tannins are responsible for anti-digestive effects as they form complexes with proteins, enzyme and essential amino acids.

1.2 GEOGRAPHICAL DISTRIBUTION OF *Camelina Sativa*

Camelina sativa originated in Germany and its cultivation spread to Central Europe (Budin et al., 1995). From the beginning of 20 th century up to the 1930's *Camelina sativa* was grown sporadically in France, Belgium, Holland, the Balkans and Russia. In 1950s camelina was still grown in Sweden and it was an important crop in the USSR (Zubr, 1997). Production of camelina declined slowly after World War II since it was difficult and expensive to hydrogenate camelina oil as other oilseed crops like soybean and canola started gaining in popularity. A few farms in Slovenia (Koros'ka region) still continue to produce Camelina (Rode, 2002).

Camelina was only known as a weed growing along with flaxseed (hence the name, "False flax") in the United states and was studied to a large extent by Robinson in Minnesota in 1987 (Putnam et al., 1993). Montana State University (MSU) Agricultural Experiment Research Centers conducted a study in 2004 on nine different oilseed crops for biofuel production and camelina emerged as a potential oilseed crop for production across Montana and the Northern Great Plains. The Camelina acreage ranged from 7000 to 20,000 acres in 2006 (McVay and Lamb, 2008) and 24,000 acres in 2007. Fig 4 shows the test sites for camelina production for the year 2008 in US and Canada by Sustainable Oils, Montana, USA.

GEOGRAPHICAL PRESENCE OF CAMELINA IN US AND CANADA

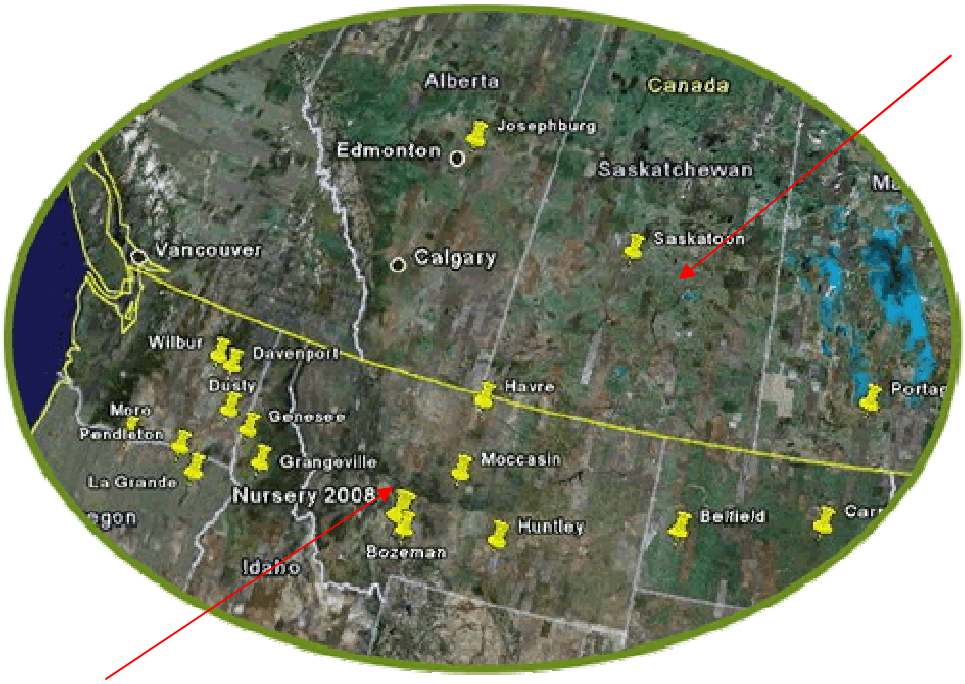


Fig 4:Camelina production test sites in Canada and USA.

(http://www.susoils.com/camelina/researchadv.php)

1.3 CAMELINA SEED PROCESSING

Vegetable oils are mostly extracted from oilseeds by mechanical pressing, by solvent extraction or a combination of both methods. Most often combinations of both methods are used for economic reasons since in oilseeds, pressing leaves a considerable amount of residual oil in the oilcakes/ meal (Gunstone, 2006).

Traditional method of pressing the camelina seeds to extract oil is still continued in Slovenia and is explained in the flow chart (Fig 5) (Rode, 2002). This method was practiced in earlier days and the oil would be sold within a few days .This was probably because the unrefined oils were being perceived as premium products and hence the oils were not refined. Zubr (1997) in his report suggested that camelina oil after extraction needs very simple refining steps such as filtration and deodorization since neutralization, degumming and bleaching of the oil were not necessary and these processing steps could adversely affect the quality of camelina Oil. Crowley (1998) reported that unrefined (cold pressed) camelina oil had an attractive yellow color but a typical mustard like aroma and flavor. Pilot scale refining of the oil was successful and the refined oil was mild in aroma and flavor and low in the levels of free fatty acids and peroxides.

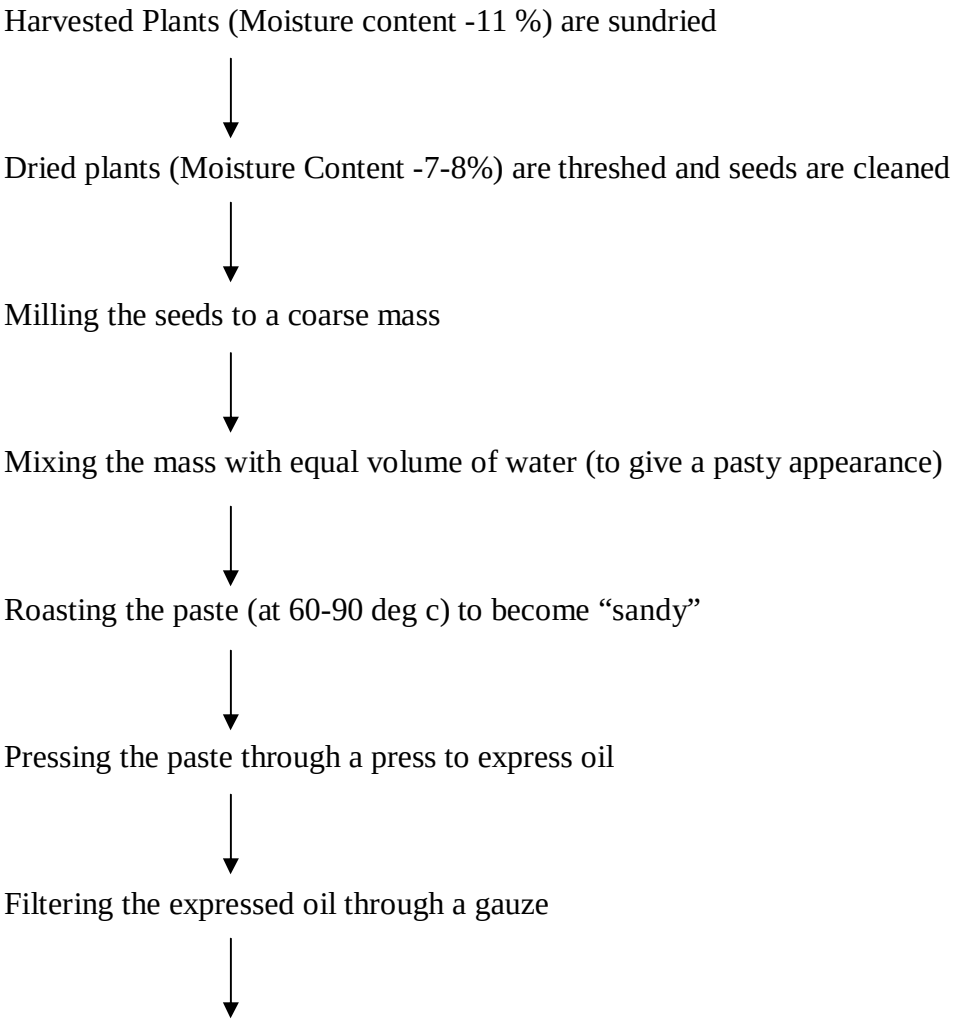


Fig 5: Traditional method of processing Camelina seeds in Slovenia

(Rode, 2002)

1.4 Refining of Vegetable Oils

Vegetable Oils can be refined by chemical or physical methods and the choice of the method depends on the oil. Refining of oils help in removing impurities such as phospholipids, free fatty acids, peroxides, polymers, pigments, metals and secondary oxidation products and also minimizes trans-fatty acid formation and tocopherol loss(Tasan et al.,2003).

Chemical refining

Industrial Chemical refining process consists of 4 steps (Belitz and Grosch, 1999, Tasan et al., 2003) and mostly done on oils which are rich in phospholipids like soybean and cottonseed oils. The latter contains gossypol which cannot be removed by the physical refining method.

1. Degumming: Degumming of the crude oil with 0.2% (of oil weight) of Phosphoric acid (85%) at 60 ° C with slow agitation for 30 minutes. This process removes the phospholipids and trace metals and the gums are separated by centrifugation.
2. Neutralization: The degummed oil is treated with 15% sodium hydroxide solution or other alkali at 80-90 ° C for 10 minutes with mild stirring. This step removes the free fatty acids, phospholipids, pigments, trace metals and sulphur compounds. The soap stock is separated from the refined oil by decanting and centrifuging. The neutralized oil is then washed with water and vacuum dried.
3. Bleaching: Bleaching of the oil is carried out using 1 % (of the oil) bleaching earth at 80 ° C for 25 minutes with vigorous stirring. The bleached oil is then

cooled to 60 ° C and filtered. This process removes the colored impurities. The oil is then winterized from 30 to 5 °C and then held at 5 °C for 10 hours

4. Deodorization: The oil is deodorized by heating it to a high temperature between 180-260 ° C under low pressure (0.5-10 mbar) for 20 minutes to 6 hours and with steam or nitrogen injection. The heated oil is then cooled to 45 ° C and this process removes the volatile oxidation products responsible for undesirable flavor and odor.

Physical Refining

Physical refining process consists of 3-4 steps and is usually done on oils with low phospholipids levels (Tasan et al., 2003).

1. Degumming: Degumming of the Crude oil with 2 % (of oil wt) water at 70 °C for 30 minutes. The degummed oil is vacuum dried and subjected to dry degumming with 0.1% citric acid (64%) at 30 °C for 30 minutes.

2. Bleaching: The degummed oil is bleached at 100 °C with vigorous stirring using 1 % activated earth (wt /wt of oil). The bleached oil is then cooled to 75 °C and filtered. The oil is winterized from 30 to 5 °C and then held at 5 °C for 10 hours.

3. Deodorizing: The winterized oil is deodorized by heating the oil to 265° deg c for 1 hour with steam injection and then cooling it to 50 °C. This process removes the free fatty acids, the mono- and di-acylglycerols and oxidation products.

1.5 Types of Camelina Oil

Camelina oil is available in two forms namely crude (or unrefined) and refined (refined, bleached and deodorized).

Crude or Unrefined Camelina Oil:

Cold mechanical pressed camelina oil is referred to as Crude, Virgin or Unrefined camelina oil. This oil is golden yellow in color and has a typical mustard kind of odor with mild nutty notes. Shelf life of unrefined camelina oil is 12-24 months without much change in flavor and aroma of the oil under ambient conditions of storage (Crowley et al., 1998). The shelf life of unrefined camelina oil depends on the storage conditions and unrefined camelina oil is more susceptible to degradation by light than by temperature (H.Abramovic and V.Abram, 2005). Natural anti-oxidants (Tocopherols) present in camelina oil could be responsible for the ambient storage stability of unrefined camelina oil.

Refined Camelina Oil:

Camelina oil was refined by the Chemical refining process and the RBD Camelina oil was colorless to pale yellow in color with a mild nutty and green, oily aroma. The shelf life of refined camelina oil was 6-9 months (Crowley, 1998). Refined Camelina oils provided in this study were cold pressed using Taby Press (Sweden) or Kolvet Press and chemically refined (degummed, neutralized, bleached, winterized and deodorized under vacuum with steam injection).

1.6 Uses and applications of Camelina seeds and oil

Camelina seeds and oil find use in various applications in the food, cosmetic, pharmaceutical and other non-food related industries. Some minor use of camelina crop includes fiber made from straws and the use of plants for ornamental and dried flower arrangements. Camelina oil does not have a GRAS status in the US but has a legal food status in France, the UK and Denmark (Zubr, 2002).

1.6.1. Camelina Seeds

Camelina seeds are mainly used for the production of oil and the by-product of oil extraction, oilseed cakes. Some other culinary uses of camelina seeds have been mentioned where the crushed seeds were used as an ingredient in bread (Zubr, 2002). Camelina seeds can also be used in the diet (up to 15%) of rabbits for the production of healthful rabbit meat and was found helpful in reducing the saturation,atherogenic and thrombogenic indexes of rabbit meat (Peiretti et al.,2007).

1.6.2. Camelina Oil:

Globally Camelina oil was used as edible oil, and for cosmetic, home remedy and industrial oil applications. Camelina oil had been used partly as edible oil and mostly as a traditional home remedy in Slovenia. The oil was considered a good remedy for stomach and duodenal ulcers, treatment of burns, wound and eye inflammations (Rode, 2002). This was probably due to the high percentage of polyunsaturated fatty acids (linolenic acid, 18:3, n-3 and linoleic acid, 18:2, n-6) which have anti-inflammatory properties. A major portion of the camelina oil produced in the US is used by the cosmetic and biofuel industries since camelina does not have a GRAS status. Camelina growers expect more

value for their crop since the n-3 fatty acid content in camelina oil makes it more suitable to the food industry than the biodiesel industry.

Uses of Camelina oil based on fatty acid composition

The fatty acid profile of camelina oil has been well established over several years by numerous researches and the oil is an excellent source of essential fatty acids (linoleic acid, 18:2, n-6 and linolenic acid, 18:3, n-3). Ingested α-linolenic acid is converted to the long chain polyunsaturated fatty acids like eicosapentaenoic (EPA) and docosahexaenoic (DHA) by elongation and desaturation enzymes (Dubois et al., 2007, Eidhin et al., 2003). These derivatives have anti-inflammatory, anti-thrombotic, anti-hypertensive and anti-arrhythmic actions. Regular cooking oils like sunflower, safflower, soybean, corn and canola oils are high in n-6 fatty acids and with the exception of canola, most of these oils have very low levels of n-3 fatty acid. The n-6 fatty acids are responsible for the inflammatory and thrombotic metabolites at high levels and hence a proper balance of the n-6 to n-3 fatty acid ratio (4:1 or 2:1) is required in the diet (Eidhin et al., 2003).

Camelina Oil can be used as in specialty oils, ω-3-enriched margarines, salad dressings and cream spreads. It can be used in foods intended for baking and shallow frying; however deep-fat frying in this oil tends to develop a strong paint-like flavor which can be carried over to the fried food (Crowley, 1998).

Vegetable oils like sunflower and soybean are rich in polyunsaturated fatty acids and are used as desirable emollients in skin care applications (Alander, 2006). The fatty acid profile of camelina makes it a suitable raw material for cosmetic products like skin creams and lotions, balms, lipsticks and bar soaps.

Uses of Camelina oil based on Unsaponifiables fraction

Phytosterols (plant sterols) are members of the “triterpene” family of natural products and more than 100 different plant sterols have been identified. Although most plant sources contain very little or no cholesterol, cholesterol has been reported in camelina oil in the order of 188 ppm (Shukla et al., 2002). Phytosterols abundant in nature are sitosterol, campesterol and stigmasterol (Belitz and Grosch, 1999). They are of nutritional interest because of their potential to lower both total serum and LDL-cholesterol in humans with high serum cholesterol by inhibiting the absorption of dietary cholesterol as well as the re-absorption of cholesterol excreted into the bile in the course of the enterohepatic cycle (Schwartz et al., 2008). The phytosterols sitosterol, campesterol and stigmasterol are abundant in camelina oil and hence the oil can be used in functional foods (like Benecol ®) and dietary supplements that help in lowering the blood cholesterol levels.

Tocopherols and phytosterols usually present in minor quantities in refined vegetable oils offer both antioxidant and bioactivity in skin care formulations (Alander et al., 2006). Tocopherols help in protecting cellular components such as DNA, proteins and lipids against free radicals and reactive oxygen species caused by exposure to harmful UV radiations and air pollutants. In the study by Alander et al., 2006, it was shown that addition of fractionated canola oil with sufficient amounts of tocopherol (710 ppm) and phytosterols (6800 ppm) to the formulation did prevent the oxidation of proteins and cell-membranes on exposure to UV rays. Camelina oil also has a total tocopherol content of 750 mg/kg oil (Abramovic et al., 2007) and phytosterols (511 mg/100 g oil) (Schwartz et al., 2008) which make it a suitable ingredient in cosmetic preparations that help in preventing photo-oxidation.

Uses of Camelina oil for biodiesel

Camelina sativa was originally test grown in Montana and the dry lands across the Midwest US as a biofuel industry raw material. Camelina demonstrated better drought tolerance and spring freezing tolerance, lower nutrient requirements and good resistance to insects and weeds than other oilseed crops. The production of biodiesel was also much cheaper than from canola or soybean oilseeds. This has led to the increased production of camelina in Montana and in 2007; 24000 acres of camelina were planted. The biodiesel produced was comparable in quality to that produced from canola and soybean (McVay and Lamb, 2008).

Uses of Camelina oil in non-food related products

Camelina oil can also be used as a drying oil because of its high Iodine number (140-160 g I_2/100 g Oil) and this property is used in oil and paint industry along with linseed oil (Zubr, 1997).

1.6.3. Camelina Meal

Camelina meal typically contains 10-15 % residual oil and 40%crude protein, minerals and 10-12 %crude fiber. Camelina meal as an animal feed additive has been studied and was found beneficial in increasing the ω-3 fatty acid content in eggs and the meat quality of laying hens and broiler chickens (Ryhanen et al., 2007, Rokka et al., 2002). Camelina meal was also used a source of ω-3 fatty acid in farmed fish, but it was reported that rainbow trout fish refused it due to the off-taste of the meal. However, rainbow trout did consume a feed formulated with camelina oil (McVay and Lamb, 2008).

2. LITERATURE REVIEW

2.1 Fatty acid profile

Oil content from camelina seeds have been reported by several research groups and can range from 25 % to 48% (Budin et al., 1995; Angelini et al., 1997; Zubr, 2003; Rode, 2002; Vollman et al., 2007; Putnam et al., 1993; Zubr, 1996). The adaptability of crops to the environment causes considerable variation in the oil content of the seeds from different locations.

Major fatty acids in camelina oil are α-linolenic (18;3,n-3), linoleic (18:2,n-6), oleic (18:1,n-9), gondoic (20:1,n-9) and palmitic(16:0) as the major fatty acids and this composition has been well confirmed by several authors (H.Abramovic et al.,2007;Budin et.al,1995;Shukla et.al,2002 ; Zubr and Matthaus,2002; Rode 2002; Putnam et al., 1993). Stearic (18:0),arachidic (20:0),eicosadienoic (20:2),eicoatrienoic (20:3), behenic (22:0), erucic (22:1),lignoceric (24:0) and nervonic acid (24:1) have also been identified in camelina oil in small quantities (Zubr and Matthaus,2002;Eidhin et al.,2005).

Interest in Camelina oil is mainly due to its polyunsaturated fatty acid content (40-50% of the total fatty acids) and low saturated fatty acid content (10-15%). Levels of α-linolenic acid (30-40%), linoleic acid (15-25%), oleic acid (10-25%), eicosenoic acid (13-18%) and erucic acid (2-5%) have been reported previously (Zubr 2002, Putnam et al., 1993). Variations in the level of α-linolenic acid have been observed in camelina oil produced in different locations in Europe and the United States (H.Abramovic and V.Abram, 2005). Similarly variations in the levels of linoleic, oleic,eicosenoic and erucic acids have also been reported(Zubr and Mathaus,2002) and it can be understood that the chemical composition of camelina oil is greatly influenced by the effects of cultivar

variety and uncontrollable factors such as the quality of soil and climatic and weather conditions (Zubr and Matthaus,2002). It has also been reported that in oilseed crops, the level of polyunsaturated fatty acids in general is promoted by low temperatures (winter and spring season) during the seed filling period, while at higher temperatures (summer season) the concentration of saturated fatty acids is enhanced (Vollman et al.,2007).

Triacylglycerol (TAG) structures are oil specific and are closely related to the fatty acid composition of the vegetable oil. The determination of TAG structures by Mass Spectrometry directly from camelina oil had revealed the presence of 50 individual TAGs. The major TAGs identified were LnLnP, LnOP, LnLnL, LnLnO, LnLnS, LnLS, LOO, LnLnG, LnLG and LLG. (Kirst et al, 2006).

2.2 Unsaponifiables fraction

Tocopherols (T) and tocotrienols (T3) present in Camelina oil have been reported in several studies (Abramovic et al.,2007,Budin et al.,1995,Zubr and Matthaus,2002, and H. Abramovic and V.Abram ,2005). Gamma-tocopherol (14.30 mg/100 g seed) was the major isomer followed by α-tocopherol (1.75 mg/100 g seed) and β-tocopherol (1.0 mg/100 g seed) (Budin et al., 1995). In this report minor levels of α-tocotrienol, β-tocotrienol and δ-tocopherol were also identified. In the study by Zubr and Matthaus (2002), the presence of γ- tocopherol (651 to 922 ppm) and the content of α-, δ-tocopherols and P-8 chromanol to the order of 15-20 ppm were reported in camelina oil. Schwartz et al., (2008) reported values of γ-tocopherol (72 mg/100 g oil), α-tocopherol (3.8 mg/100 g oil), δ-tocopherol (1.5 mg/100 g oil) and β-tocopherol (0.09 mg/100 g oil) in camelina oil.

Shukla et al.,(2002) reported the presence of cholesterol (188 ppm), brassicasterol (133 ppm), campesterol (893 ppm), stigmasterol (103 ppm), sitosterol (1884 ppm), δ5-avenosterol (393 ppm), cycloartenol (515 ppm) and 24-methylene cycloartenol (124 ppm) in refined camelina oil. It was also mentioned that several (10) minor components were unidentified due to very low concentrations of these compounds. The unsaponifiables matter was 0.54% of the oil and the levels of the major sterols were comparable to that found in rapeseed and mustard oil. Schwartz et al.,(2008) studied the plant sterol content (mg/100 g oil) of 14 vegetable oils in Finland and reported campestanol (1.6 mg), sitostanol (2.5 mg), stigmasta-5,24-dienol(6.2 mg), gramisterol+α-amyrin (1.9 mg),δ7-avenosterol (trace levels) and citrostadienol (1.30 mg) in camelina oil in addition to the sterols identified by Shukla et al.,(2002). They reported the levels of the common sterols (mg/100 g oil) in camelina oil as cholesterol (35 mg), brassicasterol (27 mg), campesterol (117 mg), stigmasterol (5.6 mg),sitosterol (300 mg), δ5-avenosterol (37 mg), cycloartenol (10 mg) and 24-methylene cycloartenol (1.0mg). Cholesterol in camelina oil was higher than most vegetable oils in this study and brassicasterol is a unique sterol found in the Brassicaceae family.

2.3 Flavor and Aroma compounds

Up to now there is only one study done by Kirst et al., (2006) on the volatile and semi-volatile compounds found in camelina oil. Their study was done on the headspace volatiles of camelina oil to understand the compounds that were responsible for the unique aroma of this oil and use the data as a basic tool for quality assurance. The study was done using Solid Phase Micro Extraction (SPME) method for collecting the

headspace volatiles at room temperature and the compounds were identified using GC-MS. This method also tried to eliminate the flavor-active degradation products formed during the heating of the sample since the study was carried out at room temperature. The volatiles were separated and identified using a GC-MS with an RTx-5 (Restec) non-polar column (60m×0.25 mm i.d and film thickness 0.25 μm). The results showed that odor profile of camelina oil was based on flavor-active degradation products (like acetic, butyric and isovaleric acid) and odor-active degradation products (like aldehydes and ketones). 31 volatile compounds was identified in their study and trans-2-butenal, acetic acid, trans, trans-2, 4-heptadienal and trans, trans-3, 5-octadien-2-one were identified as the major compounds in the headspace volatiles. The aroma of camelina oil was described as green, grassy odor with lemony notes on organoleptic evaluation.

3. HYPOTHESIS AND OBJECTIVES OF RESEARCH

3.1 Hypothesis of the research

Characterization of the fatty acids and identification of unsaponifiables matter in camelina oils grown across Montana will help us in understanding the influences of geographical conditions (like location and weather pattern) and oil processing methods on the chemical quality of the oil. Levels of fatty acids like erucic and eicosenoic acids in camelina oil produced in Montana can be used towards the FDA-GRAS petition. Eicosenoic acid is believed to be a precursor in the metabolism of erucic acid which has been implicated with myocardial lipidosis and necrosis of the heart muscles in rat model studies (Zubr, 2002).

Literature review on the volatiles found in camelina oil showed that very little work has been done in this front and only one work has identified the aroma compounds in camelina oil headspace (Kirst et al., 2006). The camelina oils provided in our study had a typical green, earthy (bell-pepper type) aroma with mild mustard notes which made it different from the aroma profile of the camelina oil analyzed by Kirst et al., 2006. Hence the use of dynamic headspace (purge-and-trap) method at 150 °C will enable us to identify most of the compounds that could be responsible for the unique aroma of camelina oil. The analysis of headspace volatiles from camelina seeds will help us in identifying the volatile compounds that can contribute to the aroma of camelina meal after the extraction of oil. Camelina seeds for volatile analysis included intact seeds, camelina meal and camelina seeds that were preheated and crushed.

3.2 Objectives of the research

The objectives of this study were to identify the volatile and semi-volatile compounds responsible for the unique aroma of camelina oil and also determine the fatty acids and unsaponifiable components of this oil.

Chemical characteristics to be studied were:

1. Fatty acid composition: Characterize the fatty acids present in (18) camelina oil samples using GC-FID and GC-MS and use this information towards the GRAS status petition.

2. Chemical composition of unsaponifiables matter: Identify the unsaponifiable compounds present in 8 Camelina oil samples using GC-MS.

3. Volatiles and semi-volatiles aroma composition: Identify the flavor and aroma compounds present in camelina seeds (7 samples) and oil (8 samples) using headspace methods and GC-MS.

4. Free fatty acids content: Determine the level of Free Fatty acids in 8 Camelina oil samples as a measure of hydrolytic rancidity.

4. MATERIALS AND METHODS

4.1 Characterization of fatty acids in camelina oil

The major component (94-98%) of crude and refined oils is triacylglycerol, which consists of three fatty acids attached to a glycerol backbone. The most common fatty acids found in oilseeds are stearic, oleic, linoleic, linolenic and palmitic (Hernandez, 2006). In order to characterize the fatty acids, the triglycerides in each camelina oil sample were subjected to trans-esterification, which converts the fatty acids to their respective methyl esters. The methyl esters can be easily separated and characterized using Gas Chromatography with Flame Ionization detector (GC-FID) and identified using Gas Chromatography-Mass Spectrometry (GC-MS).

4.1.1 Sample materials

Camelina oil samples used in this study are summarized in Table 1. The table lists the sample numbers, its description, origin, process details and aroma attributes. All samples were clear liquid at room temperature except camelina oil wax which had a lot of wax settling at the bottom of the container. The unrefined oils were yellow to golden yellow in color and refined oils were colorless to mild yellow color. A total of eighteen (18) camelina oil samples consisting of 3 chemically refined (degummed,neutralized, bleached,winterized and deodorized) samples, 2 deodorized samples and 13 unrefined oils samples were analyzed for fatty acid composition. All oil samples were produced from summer cultivars grown across Montana in 2007. The deodorized samples were subject to only deodorization under vacuum with steam injection, after extraction and filtration, sample GNG-12 was deodorized 3 times and sample GNG-13 was deodorized only 1 time.

Table 1: Sources, Process history and description of Camelina oil samples used in the characterization of fatty acids.

S.No	Sample Code	Sample	Process	Color	Aroma
		Description	**Condition**		
1	GNG1	Camelina Oil 1C107	Unrefined	Golden yellow	Oily, green and bell pepper
2	GNG 3	Camelina Oil M 1206	Unrefined	Golden yellow	Mild green and Oily
3	GNG-4	Camelina oil RDB 107*	Refined	Mild yellow	Oily and mild green
4	GNG-5	Camelina Oil ,Culbertson 07	Unrefined	Yellow	Oily and nutty
5	GNG-7	Camelina Oil,Sydney,04	Unrefined	Golden yellow	Oily and nutty
6	GNG-8	Camelina Oil,GF 1007	Unrefined	Golden yellow	Oily ,green bell-pepper
7	GNG-9	Camelina oil,GF 1007,filtered at Culbertson	Unrefined	Golden yellow	Oily and nutty
8	GNG 10	Camelina Oil,GF 1007	Unrefined	Orange	Oily and spicy
9	GNG-11	Camelina Oil,M 1206 ,Malta harvest,Dec 06	Unrefined	Golden yellow	Oily and green bell-pepper
10	GNG-12	GNG-1-Deodorized 3 times,Camelina Oil,10/07**	Deodorized	Yellow	Oily and mild green
11	GNG-13	GNG-1-Deodorized 1 time Camelina Oil,10/07**	Deodorized	Yellow	Green and oily
12	GNG-14wax	Camelina Oil wax(oil with heavy wax)	Unrefined	pale yellow	Oily and fatty
13	GNG-17	Camelina Oil MK 1206	Unrefined	Golden yellow	Oily, green bell-pepper
14	GNG-18	Camelina Oil M 1207	Unrefined	Golden yellow	Oily and mild green
15	GNG19	Camelina Oil C 107	Unrefined	Golden yellow	Oily, green bell-pepper
16	GNG21	Camelina Oil C 107	Unrefined	Golden yellow	Oily and mild pepper
17	GNG22wax	Camelina Oil RBD*(from bottom of container)	Refined	Pale yellow	Mild oily and green
18	GNG 22	Camelina Oil RBD*	Refined	Pale yellow	Oily and mild green

*****Refined oil samples (Chemically refined-degummed, neutralized, bleached, winterized and deodorized (under vacuum with steam injection)).

**Deodorized samples (subject only to deodorization at 230 deg C under vacuum with steam injection).

4.1.2 Chemicals and Reagents

A 0.5 N methanolic base reagent and a Fatty Acid Methyl Ester standard mix (known as Supelco 37 component FAME mix) were obtained from Supelco, Inc., (Bellfonte, PA) and were stored in a refrigerator and freezer respectively. Toluene, hexane and anhydrous Sodium sulfate were procured from Fisher Scientific Inc., (Fairlawn, NJ) and stored at room temperature.

4.1.3 Trans-esterification of fatty acids

About 10-30 mg of individual camelina oil samples were weighed into 20 ml test tubes. Each sample was solubilized with 1 ml aliquots of toluene prior to the addition of 2.0 ml aliquots of the methanolic base reagent. The test tubes were then placed in a heating block and incubated at 80 ° C for 15-20 minutes. The test tubes were capped at all times during the heating process and were agitated occasionally using a vortex genie mixer (Scientific Industries Inc., Bohemia, NY). After incubation, the test tubes were allowed to cool to room temperature and 1.0 ml aliquots of water were added to each of the test tubes followed by 1.0 ml aliquots of hexane. The water and hexane layers were allowed to separate completely and the upper organic layer was transferred with a Pasteur pipette to a 5 ml vial containing anhydrous sodium sulfate to ensure that the organic layer was free of moisture. The derivatized samples were stored in a refrigerator at 5 °C until analysis.

4.1.4 Instrumentation

GC-FID Analysis

A Varian 3400 gas chromatograph equipped with a flame ionization detector was used in the study to separate and characterize the fatty acid esters. The column used was a

high performance capillary column, DB-Wax, 60 m × 0.32 mm × 0.15 µm film thickness (Agilent, Hewlett Packard, Wilmington, DE). The carrier gas was helium at a flow rate of 1 ml/minute, head pressure was 20 psi and the split ratio used was 1:100. The injector and detector temperature were set to 240° C and 250°C respectively. The column initial temperature was held at 40°C for 3 minutes and then increased to 240°C at the rate of 10°C /minute with a final hold for 20 minutes. Chromatograms were recorded and processed using a Peak Simple Chromatographic data system (SRI Instruments, Torrance, CA). The total time taken to run one sample was 60 minutes. Characterization of the fatty acid components was achieved with the Supelco FAME mix standard and their identities confirmed by GC-MS (described below). GC-FID was used to separate, characterize and quantify the fatty acids present in all camelina oil samples.

GC-MS analysis

For GC-MS analysis, a HP-Wax capillary column with dimensions similar to the column used in GC-FID analysis of fatty acid components of camelina oil was used. The carrier gas was helium at a flow rate of 1 ml/minute and head pressure was 20 psi. The split ratio used here was 1:50 and the injector temperature was set at 240°C. The column initial temperature was held at 40 °C for 5 minutes and then allowed to increase to 240 °C at the rate of 10°C /minute with final hold of 20 minutes. The end of the GC capillary column was inserted directly into the ion source of a Finnigan MAT 8230 high resolution, double focusing magnetic sector mass spectrometer via a heated transfer line maintained at 240° C. The mass spectrometer was operated in an electron ionization mode(EI) ,scanning masses 35-450 amu once each 0.6 second with a 0.8 second interscan time. The mass-spectrometric data were acquired and processed with Finnigan MAT SS

300 data system. The identification of the fatty acids was carried out using the National Institute of Standards and Technology (NIST) Mass spectral library. A total of 3 camelina oil samples (GNG-1, GNG-9 & GNG-13) were selected to confirm the identities of fatty acids using GC-MS. These 3 samples were chosen as GC-FID results revealed the presence of typical fatty acids based on the FAMES standard mix used for characterization.

4.2 Unsaponifiables Fraction extraction and identification

The unsaponifiables content in each camelina oil sample was determined using the official method of Analysis 20.081 of the Association of Official Analytical Chemists (AOAC William Horowitz, 1980) with slight modifications to extract and obtain the unsaponifiable fraction. The fractions were then dissolved in methylene chloride before the individual components were separated and identified by GC-MS.

4.2.1 Sample materials

A total of eight samples (8) camelina oil samples containing 4 unrefined, 2 deodorized and 2 refined samples were analyzed for their unsaponifiables content. The samples used in this study are listed below.

1. GNG -19 C 107, unrefined camelina Oil
2. GNG- 18 M 1207, unrefined camelina Oil
3. GNG-1C (107), unrefined camelina oil
4. GNG- 17 MK 1206, unrefined camelina oil
5. GNG-12, deodorized camelina oil (deodorized 3 times)
6. GNG-13, deodorized camelina oil (deodorized 1 time)
7. GNG-4 RBD, refined camelina Oil
8. GNG-22 RBD, refined camelina Oil.

4.2.2 Chemical reagents

A 200 proof ethyl alcohol was obtained from Pharmco Products Inc., (Brookfield, CT). Ethyl ether; methylene chloride and acetone were procured from Fisher Scientific Inc., (Fairlawn, NJ). Potassium Hydroxide (KOH) flakes were obtained from Acros Organics (NJ). Concentrated KOH solution was made by dissolving 60 g KOH in 40 ml

of water and a 0.5 N KOH solution was made by dissolving 28 g of KOH in 1 liter of water.

4.2.3 Isolation and characterization of Unsaponifiables fraction in camelina oil.

Each Camelina oil sample was accurately weighed (0.2-0.25 g) into a 50 ml test-tube (A) and 2.5 ml of alcohol and 150 µl of concentrated KOH solution were added into it. The test tubes were capped tightly and placed on a heating block at 100 °C for 1 hour to saponify the samples. Samples were agitated occasionally using a vortex genie mixer and care was taken to avoid any loss of alcohol during the saponification process. While the solution was still warm, 5 ml of water was added followed by 5 ml of ethyl ether. The samples were shaken vigorously and the ether and water layers were allowed to separate and clarify. The lower aqueous layer (soap solution) was carefully transferred by means of a Pasteur pipette into another 50 ml test tube (B). In order to wash out all the soap in the ether layer in the test tube A, a 5 ml aliquot of water was added to test tube A and the layers were allowed to separate and clarify. The lower aqueous layer was transferred into test tube (B) and the aqueous extract in test tube (B) was extracted 4 times with 5 ml aliquots of ethyl ether. Each time, the upper ether layer including the emulsion layer in test tube B was transferred and pooled into test tube A. The combined ether extracts in test tube A was then washed 3 times with 5 ml aliquots of water and the ether-water mixture was gently shaken to avoid emulsion formation. Each time the layers were allowed to separate and clarify, and the aqueous layer was removed carefully without disturbing the emulsion layer. Following the water wash, the ether layer in test tube A was subjected to 3 alternate washes of 5 ml portions of 0.5 N KOH solution and 5 ml aliquots of water. The mixture was thoroughly shaken and the layers were separated by

centrifugation at 2000 rpm for 15 minutes. During washing, whenever an in-between layer of emulsion was formed as much as the aqueous layer was removed leaving the emulsion with ether layer. After the 3 rd KOH treatment, the ether layer was washed successfully with 5 ml portions of water until litmus testing of wash water was neutral. The ether layer in test tube A was evaporated to about 5 ml at 30 °C under nitrogen and saved for unsaponifiables fraction determination.

Determination of unsaponifiables fraction

The ether fractions obtained by the above process were transferred to pre-weighted Fisher scientific flat bottom 44 ml disposable aluminum dishes (pre-dried in an oven at 100 °C for 30 minutes). The ether layer was evaporated off under N_2 at 30 ° C and the dishes were then held in an oven at 100 °C for 30 minutes to remove residual moisture. After cooling to room temperature, the dishes were re-weighed to obtain the weights of unsaponifiables fraction.

The unsaponifiables fractions were then dissolved with 1 ml aliquots of methylene chloride and transferred into individual 4 ml vials, capped and stored in a refrigerator for further identification by GC-MS.

Identification of unsaponifiable components using GC-MS analysis

The GC-MS used for the identification of fatty acids was also used in the analysis of the unsaponifiable fraction in camelina oil. Separation of the unsaponifiable fraction components was carried out using a HP-5 MS column (30 m × 0.25 mm × 0.25μm) (Agilent technologies, Hewlett Packard, Wilmington, DE). The carrier gas was helium with a head pressure of 15 psi and the chromatographic run was started split less and split (1:100) opened after 0.5 minutes. The injector temperature was 100 °C and the heated

transfer line was maintained at 320 °C. The column temperature was held at 100 °C for 3 minutes and then allowed to increase at the rate 10 °C/minute to 320 ° C for 90 minutes. 1.0µl of each unsaponifiables extract sample was injected for separation and identification. The total time taken to run one sample was 50 minutes and the mass range was 35-650 amu. The mass spectrometric data were acquired and processed as explained under instrumentation using GC-MS (section 4.1.4).

4.3 Free Fatty Acids

Free fatty acids indicate the extent of lipolysis in oils and fats. Hydrolysis takes place at the junction of the fatty acids and the glycerol portion of the triglyceride, resulting in glycerol and free fatty acids (Belitz and Grosch, 1999).

4.3.1 Sample materials

Eight camelina oil samples (4 unrefined, 2 deodorized and 2 refined oil samples) were used in the determination of free fatty acids in edible oil. The samples used for this study were the same set of 8 samples used in determination of unsaponifiables content (section 4.2.1).

4.3.2 Chemical reagents

Phenolphthalein (1 % in methanol v/v)**,** 0.1 N Sodium hydroxide and 0.25 N Sodium hydroxide solutions were purchased from Fischer-Scientific (Fairlawn, NJ). Ethyl alcohol (200 proof) was procured from Pharmco Products Inc, (Brookfield, CT).

4.3.3 Determination of Free fatty acids

Free fatty acids in crude and refined oils were determined by the AOAC method (Association of Official Analytical Chemists) 28.029. This method measures the amounts of sodium hydroxide that is required to neutralize the acids that are formed during oil extraction and refining processes.

Analysis of free fatty acids

7.05 gms of the unrefined oil sample was weighed into a 250 ml conical flask. To this 50 ml alcohol and 2 ml of Phenolphthalein solutions were added and 0.1-0.2 ml of 0.1 N Sodium hydroxide solution was added to produce a temporary faint pink color. This solution was titrated against a 0.25 N Sodium Hydroxide solution with vigorous

shaking until pale permanent pink appears and persists more than 1 minute. The titer value of Sodium hydroxide (ml of 0.25 N NaOH) corresponds to the % free fatty acids (expressed as oleic acid). The deodorized samples were analyzed with the same procedure followed for determining free fatty acids in unrefined oils.

The free fatty acids estimation in refined oils was slightly different than the procedure followed for crude or unrefined oils. To approximately 50 ml of ethyl alcohol in a clean dry 250 ml conical flask, few drops of the refined oil sample and 2 ml of phenolphthalein solutions were added and placed in a water bath maintained 60-65 °C. The flask was placed in the water bath till the contents in the flask was warm and to this a few drops (0.1 to 0.15 ml) of 0.1 N NaOH was added to produce a faint pink color which disappeared in a few minutes. To the weighed refined oil sample (56.4 gms) the neutralized alcohol was added and the mixture was titrated against 0.1 N NaOH solution. The solution was titrated with occasional warming and vigorous shaking until the same permanent pink appeared in the supernatant alcohol. The titer value (ml of 0.1 N NaOH) was multiplied by 0.05 and reported as % free fatty acid (expressed as oleic acid).

4.4 Volatiles and semi-volatiles

Volatile and semi-volatile compounds in camelina seeds and oil were identified using purge-and trap and static headspace methodologies.

4.4.1 Volatiles identification using Purge & Trap Thermal desorption method

The Short Path Thermal Desorption technique involves directly desorbing the volatiles and semi-volatiles collected on an adsorbent resin in a small sample tube onto the injection port of the gas chromatograph. This technique can be used in the analysis of volatile and semi-volatile compounds in samples with high and low moisture content.

The purge and trap technique involves purging inert gas (mostly nitrogen) through the sample and the volatiles and semi-volatiles are collected in a glass lined stainless steel tube (GLT). These sample tubes are 3 and 4 mm internal diameter and are packed with a porous polymer resin such as 2, 6 diphenyl-*p*-phenylene oxide which is sold under the trademark Tenax-TA ™ or activated graphitized carbon sold under trademark Carbotrap ™. Both the trapping agents have a high affinity for non polar organic compounds and a very low affinity for water vapor and low molecular weight polar compounds with less than three carbon atom (Hartman *et al*., 1991; Hartman *et al*., 1993). In this study we used Tenax -TA for collection of volatiles from seed and oil samples.

4.4.1.1 Sample materials

Camelina Oil samples

A total of eight (8) camelina oil samples (4 unrefined, 2 deodorized and 2 refined oils) were analyzed for volatile and semi-volatiles study. The samples used in the volatile and semi-volatile were the same samples used for unsaponifiables extract and free fatty acids estimation (section 4.2.1).

Camelina seed samples

A total of six (6) camelina seed and one (1) camelina meal samples were analyzed for volatile and semi-volatile compounds using purge and trap method. The details of the samples are given in Table 2

Table 2: Samples of camelina seeds and meal used in volatile and semi-volatile compounds identification.

S.No	Sample Description	Type	Color	Aroma*
1	Ligena 1 seeds	Seeds	Brown	Sulfury
2	Ligena 1A seeds	Seeds	Brown	Sulfury
3	Calena 2 seeds	Seeds	Brown	Mild sulfury
4	Calena 2A seeds	Seeds	Brown	Mild sulfury
5	GNG-Omega meal	Meal	Dark brown	Cabbage & mustard type
6	Ligena 1 Crushed	Crushed seeds	Dark brown	Mustard type
7	Ligena 1Preheated	Preheated & crushed	Dark brown	Strong cabbage note

*Aroma-of the seeds evaluated based on the outgas products from tenax trap

4.4.1.2 Other materials

Desorption tubes containing 60-80 mesh Tenax adsorbent were prepared. Silanized glass lined stainless steel desorption tubes (3.0 mm i.d. × 10 cm length) from Scientific Instrument Services Inc(SIS Ringoes,NJ) were packed with a 4 cm bed volume of Tenax-TA adsorbent between plugs of silanized glass wool. The tubes were conditioned by passing nitrogen through them at a rate of 30-40 ml /minutes while heating in a desorption tube conditioning oven made in-house. The tubes were heated at 300° C for 1 hour. The tubes were then cooled down to room temperature, capped and stored. The conditioning step was performed to ensure that the adsorbent resin was free of any volatiles prior to sample loading.

4.4.1.3 Identification of volatiles and semi-volatiles in Camelina Oil.

Initially a SIS liquid bubbler type purge-and-trap system was used to directly purge and trap the volatile and semi-volatiles from the oil but this was abandoned since some of the samples had a tendency to foam. Hence we followed the following method of collecting volatile and semi-volatile compounds from camelina oil. Each oil sample was weighed (2.50-2.80 gm) into a glass vial with Teflon-lined screw cap and mixed with an equal portion of Celite 545® diatomaceous earth (Fisher Scientific, NJ). Diatomaceous earth is a naturally occurring soft chalk-like sedimentary rock and has been used in the chemical industry as a filtration aid and an adsorbent for liquids. The sample oil and celite were mixed thoroughly to ensure uniform distribution of the oil and the oil-celite mixture was transferred completely onto a glass sample tube (12-15 inches length,0.5 inch diameter) lined with silanized glass wool at both the ends. The glass tubes were loaded onto a solid sample purge-and-trap apparatus (SIS, Ringoes, NJ) and connected to N_2 gas source at one end. The other end of the glass tube was connected to a Tenax trap (which was spiked with internal standards-Benzene-d_6, Toluene-d_8 and Naphthalene-d_8 at 10 ppm each for quantification) to collect the out-gas products. N_2 gas was purged through the sample maintained at 150°C at the rate of 50 ml/minute for 30 minutes. The charged adsorbent traps were then connected to the Short path thermal desorption system and thermally desorbed directly into the GC-MS system described below for volatile and semi-volatiles analysis. Fig 6 is an illustration of the Short Path Thermal Desorber unit used in desorbing the volatiles directly into the injection port of the gas chromatograph The tenax trap was desorbed at 260 °C for 5 minutes, injection time was 30 seconds and

initial purge time was 10 seconds. During the 5 minutes desorption, the column was held at -20 ℃ to allow the volatiles and semi-volatiles to collect at the head of the column.

Finnigan MAT 8230 mass spectrometer used for identification of fatty acids and unsaponfiables extract was also used to analyze the volatiles and semi-volatiles present in camelina oil samples. A fused silica capillary column, Equity-5, 60 m × 0.32 mm ×1.0 μm (Supleco Inc,Bellfonte,PA) was used. The carrier gas was helium with a head pressure of 20 psi and the split ratio was 1:100. The injector temperature was 250°C and the heated transfer line was maintained at 280°C. The column temperature was programmed at an initial temperature -20° C (5 minutes) to a final temperature of 280°C (for 5 minutes). An increase of temperature from -20 to 30°C at the rate of 10° C/minute, from 30° to 200° C at the rate of 5 °C/minute and a final increase from 200° to 280° C at the rate of 10° C/minute was programmed. Acquisitions started at the beginning of thermal desorption and the total time taken to run one sample was 50 minutes. The mass spectrometer scanning masses were 35-350 amu and the mass spectrometric data were acquired and processed in the same way as explained earlier in section (4.1.4).

4.4.1.4 Identification of volatiles and semi-volatiles in camelina seeds

Camelina seed samples were weighed (10 gm) and transferred to a glass sample tube (12-15 inches length) lined with silanized glass wool at both the ends. The seed samples were maintained at 100°C in the solid sample purge-and-trap apparatus for 30 minutes and volatiles were collected on to the tenax trap by purging N_2 gas at 50 ml/minute through the samples. Figure 7 shows the purge and trap apparatus set up for analyzing volatile and semi-volatile compounds in camelina seeds and oil. The charged

adsorbent traps were then connected to the Short Path thermal desorption system and thermally desorbed directly into the GC-MS system under the same conditions described for camelina oil samples.

The GC-MS was the same instrument used for the analysis of volatiles and semi-volatiles present in the camelina oil samples. Equity-5 column, common to the study on camelina oil volatiles was used .The carrier gas was helium with a head pressure of 20 psi and the split ratio was 1:100.The injector temperature was 280°C and the heated transfer line was maintained at 300 °C. The column temperature was programmed at an initial temperature -20° C (5 minutes) to a final temperature of 280°C (for 5 minutes) and the temperature increase was at the rate of 10° C/minute. Acquisitions started at the beginning of thermal desorption and the mass spectrometer scanning masses were 35-350 amu. The data was processed the same way explained in section 4.1.4. Identification of volatiles and semi-volatiles were based on National Institute of Standard and Technology (NIST) Mass spectral library, published literature and reference books on retention times of compounds.

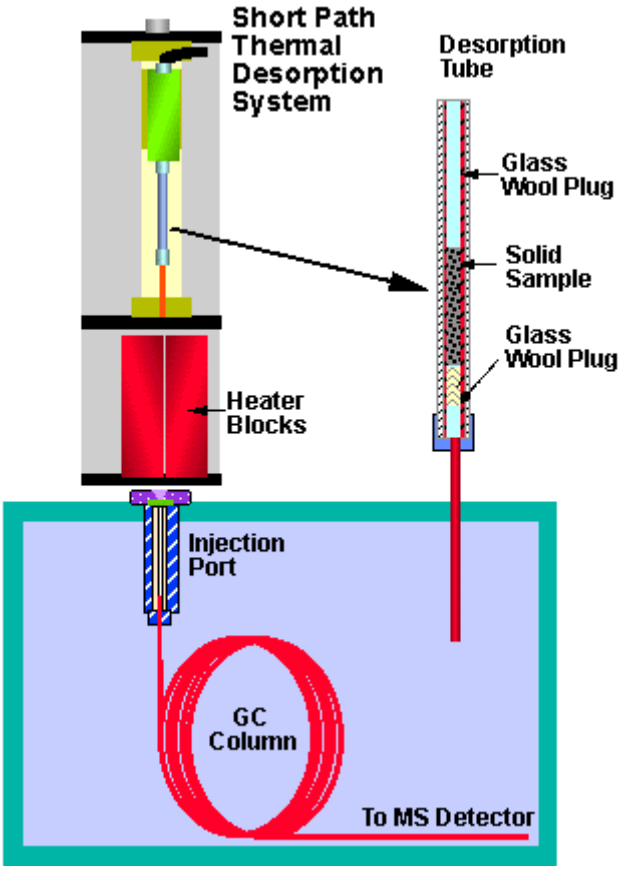


Figure 6: Short Path Thermal Desorption system

(Scientific Instrument Services Inc., Ringoes, http://www.sisweb.com/sptd/sptddesc.htm)

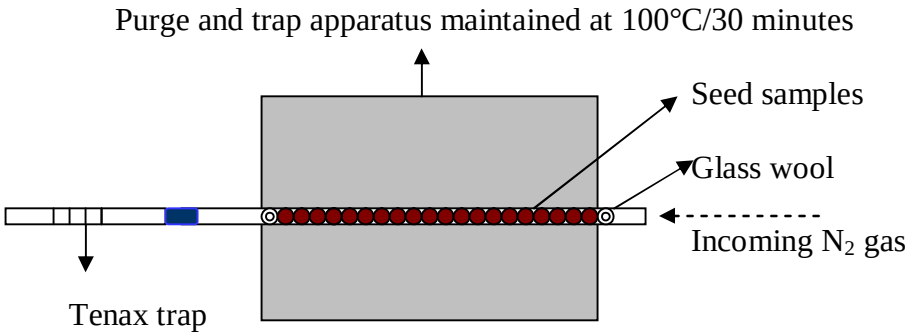


Figure 7: Schematic diagram of solid sample purge-and-trap apparatus for volatiles and semi-volatiles analysis in camelina seeds.

4.4.2 Static Headspace volatile method

Cruciferous oilseeds contain aromatic and glucosinolate compounds (Zubr, 1997) and some of the volatile compounds identified in cruciferous oilseeds like mustard are low molecular weight compounds. Static headspace volatiles method is a simple and rapid method and the sample aroma compounds that are most important to sensory perception (or in other words only the major components) are identified (Reiniccus, 1985).

Camelina seed samples (refer Table 2) were weighed (3-5 gm) and transferred to a 4 ml headspace vial and sealed with inner Teflon lined septum. The headspace vial was matrix spiked with internal standards (Benzene-d_6, Toluene-d_8 and Naphthalene d_8 at 10 ppm each for quantification). The headspace vial was then placed in an oven maintained at 100° C for 30 minutes and 1 ml of headspace volatiles was withdrawn into an air-tight syringe and injected into the GC-MS.

The GC-MS was the same instrument used for all analysis in this study and the same column (Equity-5) was used for separation of volatiles and semi-volatiles in camelina seeds. The sample was injected in a split less mode and after 0.50 minutes the split was opened. The split ratio was 1:10 and the injector temperature was 250°C. The heated transfer line was maintained at 280°C. The column temperature was programmed at an initial temperature -20° C (3 minutes) to a final temperature of 280°C at a temperature increase rate of 10° C/minute. Scanning masses were 35-350 amu and the mass spectrometric data were acquired and processed in the same way explained earlier in section 4.1.4.

5. RESULTS AND DISCUSSION

5.1 Fatty Acid Composition

5.1.1 Fatty acid methyl esters (FAMES) standard components

Fatty acids in the supleco FAME 37 standard mix were identified using GC-MS and fatty acids starting from butyric(C: 4) to nervonic (C 24:0) were identified. The fatty acids identified in supleco FAME 37 component mix using GC-FID is shown in Figure 8. Temperature program on GC-FID for the FAME 37 component mix and all camelina oil samples was maintained the same and this helped in determining a Relative Retention Time (RRT) for various fatty acids. By comparing the RRT of standard fatty acids with the fatty acids in camelina oil samples, the characterization of the individual fatty acids in camelina oil was achieved. The fatty acids present in camelina oil sample # GNG 12 is shown in Figure 9 and the fatty acids identified in sample GNG-1 using GC-MS is shown in Figure 10.

5.1.2 Fatty acids in Camelina oil

A total of 21 fatty acids were found in camelina samples. There were 10 saturated fatty acids (SFA): myristic acid (C14:0), pentadecanoic acid (C15:0), palmitic acid (C16:0), heptadecanoic acid (C17:0), stearic acid (C18:0), arachidic acid (C20:0), heneicosanoic acid (C21:0), behenic acid (C22:0), tricosanoic acid (C23:0) and lignoceric acid (C24:0). Samples contained 11 unsaturated fatty acids (USFA) out of which 6 were monounsaturated fatty acids (MUFA): palmitoleic acid (C16:1), cis-10-heptadecenoic acid (C17:1), oleic acid (C18:1), eicosenoic acid (C20:1), erucic acid (C22:1), nervonic acid (C24:1). The 5 polyunsaturated fatty acids found in camelina oil were linoleic acid(C18:2,n-6),α-linolenic acid (C18:3,n-3),cis-11,14-eicosadienoic acid

(C20:2),eicosatrienoic acid (C20:3) and cis-13,16 docosadienoic acid (C22:2). The results of fatty acids in 18 Camelina oil samples are summarized in Table 3.

The five major fatty acids found in all Camelina oil samples were α-linolenic acid (8.4-27.3 %),linoleic acid (10.14 -24.3%),oleic acid (10.1-53%), eicosenoic acid (3.81-15.3 %) and palmitic acid (5-14.12 %). In most of the samples, the poly-unsaturated fatty acids (linoleic and α-linolenic acid) were present up to 40-48% , monounsaturated (32-37%) and palmitic acid (5-14%) of total fatty acids in camelina oil with the exception of one sample, GNG-7. This sample was found to contain a very high level of oleic acid (54%) and the levels of linoleic and α-linolenic acids were 15 and 9 % respectively. This oil sample was produced from camelina harvested in 2004(Sydney, Montana) and the variations in the major fatty acids could be due to growing location since no other sample was available from this location. The levels of oleic, linoleic, α-linolenic, palmitic and eicosenoic acids in samples # GNG-1, 3, 5, 8, 9,10, 11, 4 and 22 were comparable to the levels reported by Putnam et al., (1993), Budin et al., (1995) and Angelini et al., (1997).

Stearic (3-5%), arachidic (2-7%), eicosadienoic acid (2-5%), behenic (0.2-3.7%) and erucic acid (0.3-5.10 %) were present in moderate levels in all samples. Overall ten fatty acids were present in all samples.

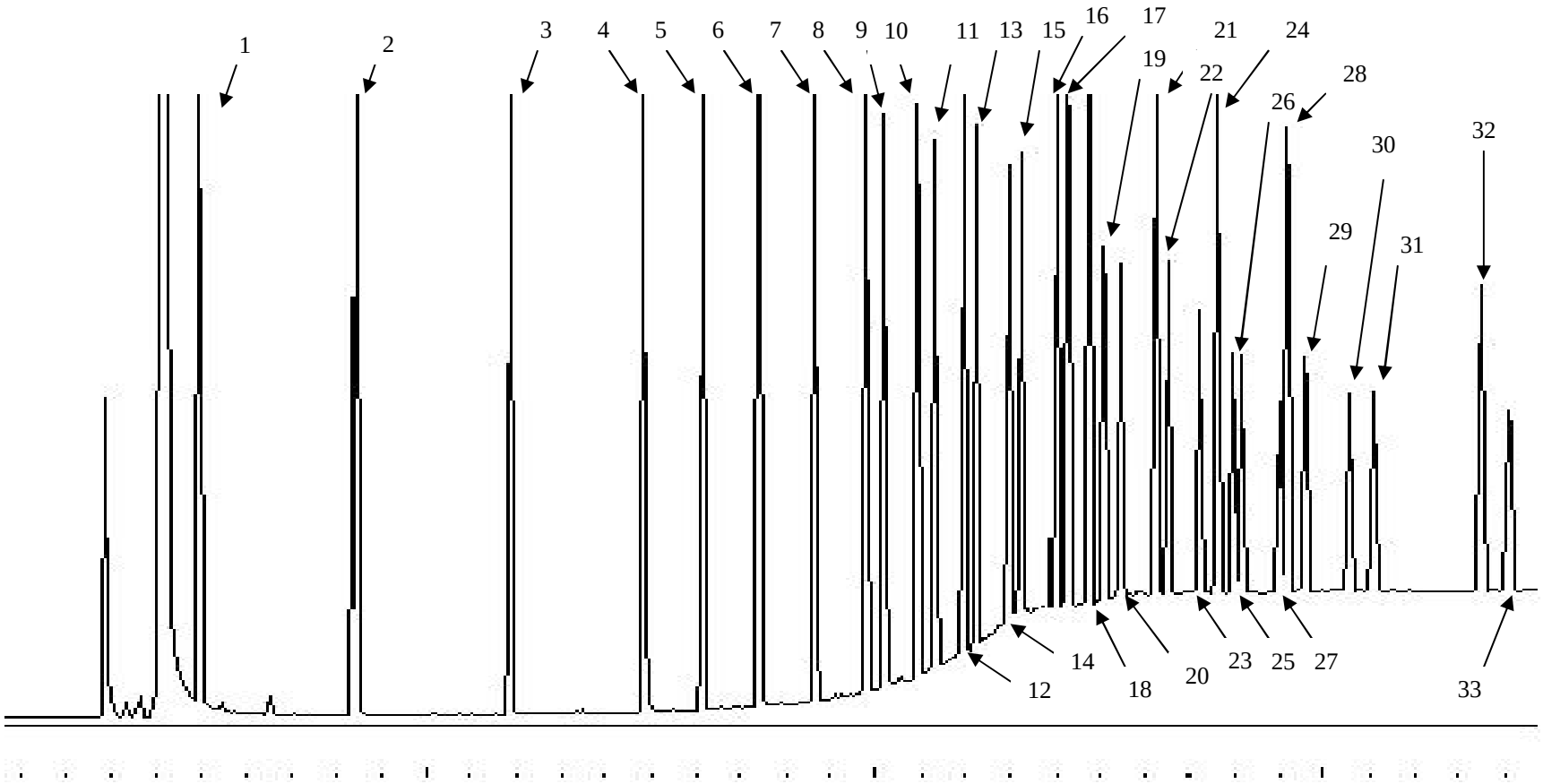


Figure 8: GC-FID Chromatogram of Fatty Acid Methyl Esters (FAMES) Standard. Methyl esters of 1.Butyric, 2.Caproic,3.Caprylic,4.Capric,5.Undecanoic,6.Lauric,7.Tridecanoic,8.Myristic,9.Myristoleic,10.Pentadecanoic,11.cis-10-Pentadecenoic,12.Palmitic,13.Palmitoleic,14.Heptadecanoic,15.cis-10-Heptadecenoic,16.Stearic,17.Oleic,18.Linoleic,19.γ –Linolenic,20.α – Linolenic,21.Arachidic,22.cis-11-Eicosenoic,23.cis-11,14-Eicosadienoic, 24.Heneicosanoic, 25.Arachidonic, 26.Eicosatrienoic,27.Eicosapentaenoic ,28.Behenic,29.Erucic,30.cis-13,16-Docosadienoic,31.Tricosanoic,32.Lignoceric,33.Nervonic

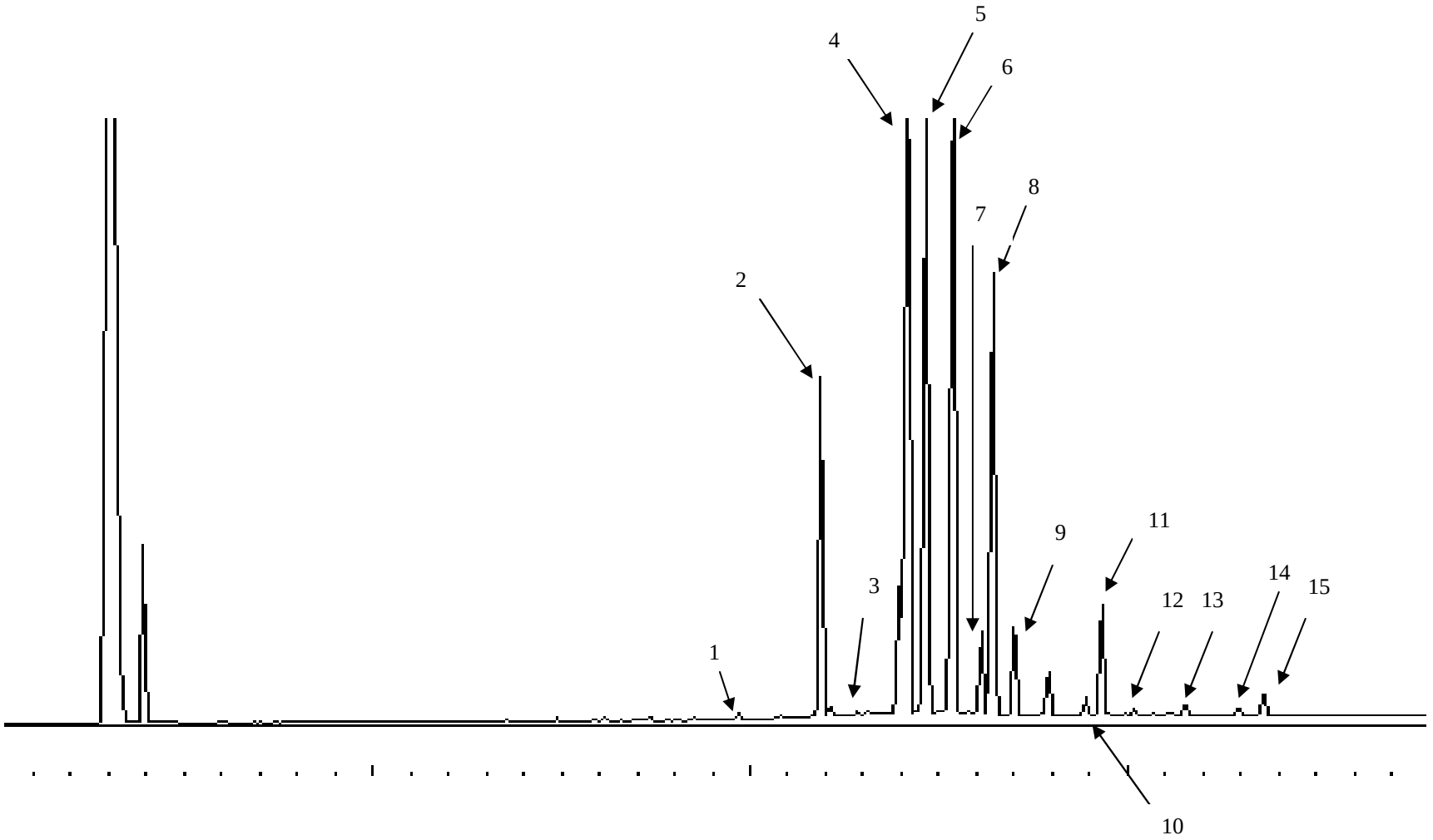


Figure 9 : GC-FID Chromatogram of fatty acids characterized in sample # Camelina deodorized oil GNG-12.Methyl esters of 1.Myristic,2.Palmitic,3.Palmitoleic,4.Stearic+Oleic,5.Linoleic,6.α-Linolenic ,7.Arachidic, 8.Eicosaenoic, 9.Eicosadienoic, 10.Eicosatrienoic,11.Behenic,12.Erucic,13.Docosadienoic,14.Tricosanoic,15.Lignoceric acid.

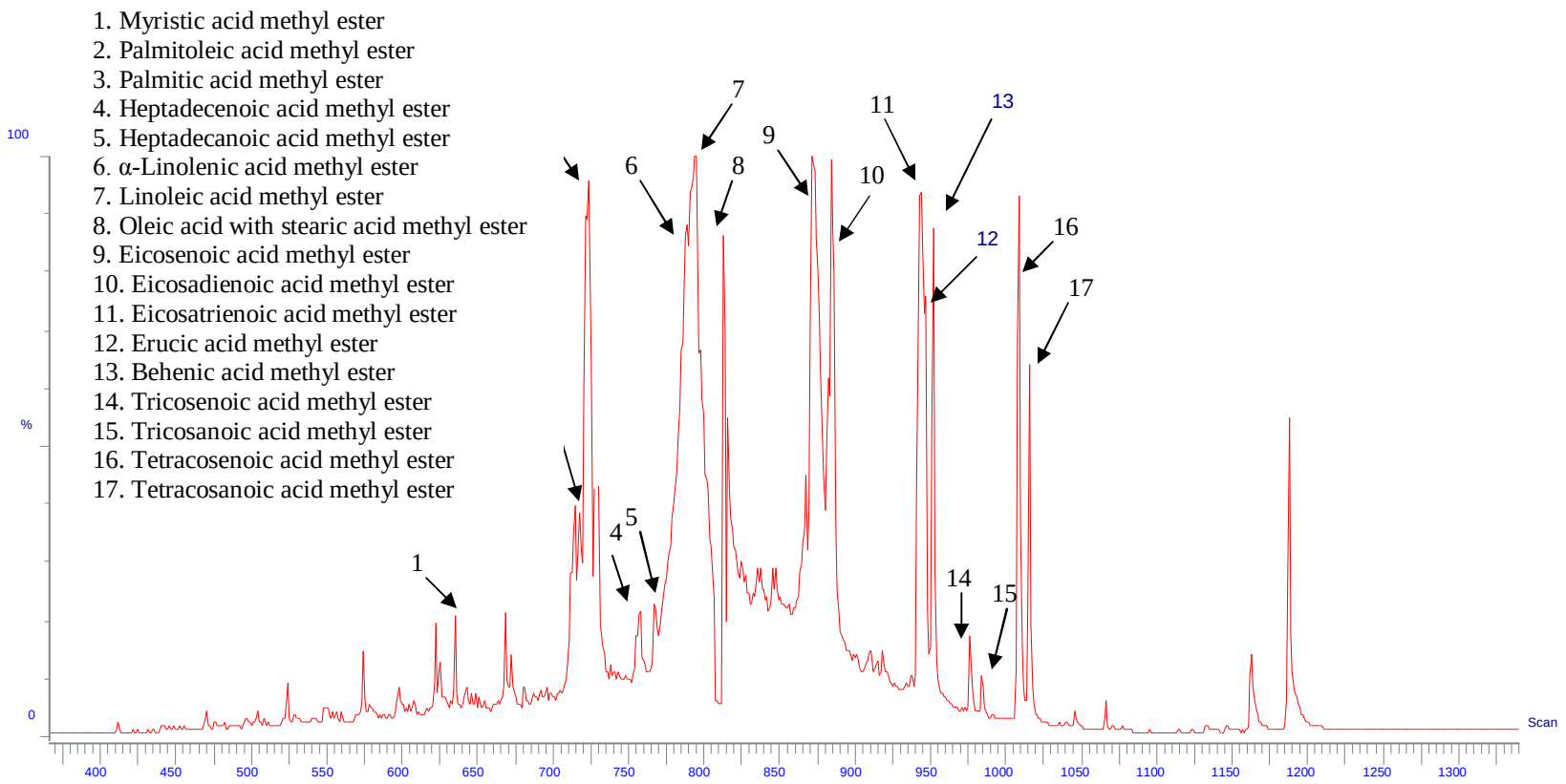


Figure 10: GC-MS chromatogram with identifications of Fatty acid methyl esters of Camelina Oil GNG-1

Other minor fatty acids in camelina oil:

Myristic acid (C14:0) was found in 3 samples (GNG-4, 12 and 13) and pentadecanoic acid (C15:0) was present only in 2 samples (sample GNG-4 and GNG-13). Myristic and pentadecanoic acids were identified only in deodorized and refined oil samples. Fatty acids like palmitoleic (C16:1), heneiosanoic acid (C21:0), eicosatrienoic acid (C20:3), cis-13, 16 docosadienoic acid (C22:2), tricosanoic acid (C23:0) and lignoceric acid (C24:0) were present in most samples in small to trace quantities. Nervonic acid was found only in 3 samples(GNG-1,GNG-17 & GNG-22 Wax) in small quantities(0.5-1.8%).Only one sample # GNG-21 had medium levels (5-7%) of arachidic acid (C20:0) and tricosanoic acid (C23:0). We believe that tricosanoic acid (C23:0) found in our study has not been identified in camelina oil in previous studies. The presence of the fatty acids palmitoleic acid (C16:1), arachidic (C20:0), eicosadienoic (C20:2), eicosatrienoic acid (C20:3), behenic (C22:2), docosadienoic acid (C22:2), lignoceric (C24:0) and nervonic acid (C24:1) in camelina oil have been reported by some authors (H. Abramovic and V.Abram, 2005, Zubr and Matthaus, 2002). Pentadecanoic and heptadecanoic acids were present in deodorized and unrefined camelina oil samples and these 2 fatty acids have also not been reported before. These are the odd carbon number fatty acids and are commonly found in milk and plant oils (Belitz and Grosch, 1999).

Major fatty acids in unrefined oils: In unrefined oil samples, the major fatty acids were α-linolenic, linoleic, oleic, eicosenoic and palmitic. Samples GNG-11 and 14wax did not follow this pattern and the major fatty acid was linoleic followed by α-linolenic, oleic, eicosenoic and palmitic acids. Erucic acid levels were found between 0.18-5.1 %.

Samples GNG-3, 5 and 14wax had very low erucic acid levels (0.15- 0.4%). Other interesting observations in the unrefined oil samples were:

1. Sample # GNG-18 had the highest level of palmitic acid (14.12 %),
2. Lowest level of α-linolenic acid (8.40 %) was found in sample # GNG-7.
3. Sample #GNG-5 had the highest level of eicosenoic acid (15.3%).
4. Tricosanoic acid(7.35%) was high in sample # GNG 21
5. Erucic acid was high in unrefined oil samples # GNG 19 and GNG 21.

Major fatty acids in deodorized samples: The major fatty acid was linoleic, followed by α-linolenic, oleic, eicosenoic and palmitic acids. The α-linolenic acid level in deodorized samples (GNG-12 & 13) were slightly lower than the original unrefined sample (GNG-1) and this could be due to the deodorization process since it has been shown that degradation of α-linolenic acid follows a first order reaction rate and depends on the temperature at which deodorization process is carried out (Henon et al., 1997). There were no major variations in the fatty acid profile between the two deodorized samples and this is expected since they were the deodorized versions of the original unrefined sample # GNG-1 (Table 3).

Major fatty acids in refined samples: The fatty acids in refined camelina oil samples were similar to the unrefined oil samples. Sample GNG-22 was the best source of α-linolenic acid and this sample was also a good source of linoleic, oleic and eicosenoic acids. Erucic acid was below 3 % in refined oil samples. The levels of palmitic, stearic acid and nervonic were high in sample GNG-22wax. Also the level of α-linolenic acid

was low in the refined oil sample # GNG-22 wax. Eicosenoic acid (15.3%) was high in sample GNG-4 and there were no major differences in fatty acid composition observed between refined oil samples GNG-22 and GNG-4 (Table 3). This probably suggests that the growing conditions and cultivar variety could be similar for these samples.

Overall it can be seen that camelina oil has a fatty acid profile which is high in unsaturates (83-85%) and low in saturated fatty acids (15-17%). Improvements in the fatty acid composition can be made by selecting cultivars that promote low yields of erucic acid like samples # GNG-1, 3, 4, 5, 14wax and 22. It can also be observed that the best sources for α-linolenic acid yields are from samples # GNG-1, 3, 4, 5, 9, 10 and 22. Also a strong negative correlation has been reported between linolenic and erucic acid in the oils from 91 accessions of the brassicaceae family oilseeds, which explained two basic patterns characterized by high linolenic and high erucic acid contents, respectively (F.D.Goffman et al., 1999).

Table 3: Percentage distribution of fatty acids C 14:0 to C 20:0 in Camelina oil samples

S.No	Sample Description	C14:0	C15:0	C16:0	C16:1	C17:0	C17:1	C18:0	C18:1	C18:2	C18:3	C20:0
	Unrefined				**(n-9)**				**(n-9)**	**(n-6)**	**(n-3)**	
1	GNG-1 (unrefined)	…..	…..	7.27	1.61	0.511	0.73	3.90	15.00	15.55	22.85	4.14
2	GNG-3(Unrefined)	…..	…..	6.72	…..	…..	…..	3.38	19.34	21.04	23.90	2.5
3	GNG-5,(unrefined)	…..	…..	6.47	0.18	…..	…..	3.27	20.12	19.91	24.54	2.33
4	GNG-7,(unrefined)	…..	…..	4.90	0.84	0.66	0.79	4.40	52.87	14.52	8.37	1.73
5	GNG-8,(unrefined)	…..	…..	5.61	0.17	0.18	0.24	3.65	15.66	17.15	24.13	3.16
6	GNG-9,(unrefined)	…..	…..	6.37	0.19	0.18	0.52	3.88	18.67	21.58	24.30	2.23
7	GNG-10(unrefined)	…..	…..	6.56	0.2	…..	…..	3.42	18.82	21.83	25.04	2.25
8	GNG-11(unrefined)	…..	…..	9.50	0.31	…..	…..	3.51	18.99	22.19	20.30	2.60
9	GNG-14, Wax	…..	…..	6.95	…..	…..	…..	3.37	19.3	24.31	23.5	2.46
10	GNG-17,(unrefined)	…..	…..	7.67	3.58	0.87	3.67	4.08	13.8	14.82	22.03	2.14
11	GNG-18,(unrefined)	…..	…..	14.12	3.33	…..	…..	4.62	13.81	17.03	18.80	3.85
12	GNG-19,(unrefined)	…..	…..	9.40	4.44	…..	…..	4.58	10.08	10.14	16.28	5.36
13	GNG-21,(unrefined)	…..	…..	5.54	4.33	…..	3.73	4.34	11.35	11.81	16.65	7.96
	Deodorized*											
14	GNG-12(deodorized)	0.15	…..	6.72	0.20	…..	…..	3.51	20.27	22.96	21.06	2.61
15	GNG-13(deodorized)	0.110	0.059	7.28	0.24	…..	…..	3.59	20.24	22.0	21.00	2.58
	Refined**											
16	GNG-22,Wax(refined)	…..	…..	9.61	3.07	…..	…..	5.87	14.19	13.77	16.62	5.11
17	GNG-22(RBD)	…..	…..	6.27	…..	…..	…..	3.04	19.25	20.6	27.31	2.2
18	GNG 4(RBD)	0.14	0.14	6.21	0.31	0.2	0.51	3.23	18.31	19.88	24.7	2.35

* Deodorized samples (GNG-12-deodorized 3 times, GNG-13-deodorized 1 time)

**Refined samples (chemically refined samples-degummed, neutralized, bleached, winterized and deodorized)

Table 3.cotd: Percentage distribution of fatty acids C 20:1 to C 24:1 in Camelina oil samples

S.No	Sample Description	C20:1	C20:2	C21:0	C20:3	C22:0	C22:1	C22:2	C23:0	C24:0	C24:1
	Unrefined	**(n-9)**					**(n-9)**				**(n-9)**
1	GNG-1C107 (unrefined)	13.36	2.91	1.63	1.57	3.77	1.56	1.50	0.50	1.15	0.54
2	GNG-3(Unrefined)	14.26	1.85	1.20	0.44	3.09	0.37	0.42	0.50	0.90	…..
3	GNG-5,(unrefined)	15.04	1.83	0.72	0.74	3.19	0.18	0.22	0.53	0.79	…..
4	GNG-7,(unrefined)	3.76	1.92	0.77	0.42	1.13	0.48	1.65	…..	0.79	…..
5	GNG-8,(unrefined)	12.86	2.66	1.86	2.04	2.33	2.33	1.62	3.15	0.89	…..
6	GNG-9,(unrefined)	14.16	1.90	0.60	0.14	0.65	2.92	0.29	0.45	0.85	…..
7	GNG-10(unrefined)	14.08	1.91	0.69	0.18	0.17	2.93	0.51	0.5	0.91	…..
8	GNG-11(unrefined)	13.55	2.44	0.93	0.74	0.21	2.86	0.75	0.43	0.87	…..
9	GNG-14 Wax	12.79	1.8	0.65	0.34	2.62	0.29	0.39	0.33	0.94	…..
10	GNG-17(unrefined)	9.91	3.83	1.00	3.2	2.24	2.9	0.5	2.35	0.82	0.51
11	GNG-18,(unrefined)	9.70	3.05	…..	2.49	2.97	3.58	…..	…..	2.36	…..
12	GNG-19,(unrefined)	12.80	5.23	5.53	4.28	3.45	5.30	…..	…..	2.83	…..
13	GNG-21,(unrefined)	12.58	5.88	…..	…..	3.22	5.10	…..	7.58	…..	…..
	Deodorized*										
14	GNG-12,(deodorized)	14.57	2.52	0.57	0.19	0.19	3.045	0.2	0.37	0.90	…..
15	GNG-13-(deodorized)	14.66	2.66	0.29	0.48	0.27	3.04	0.22	0.37	0.92	…..
	Refined**										
16	GNG-22Wax(refined)	13.1	4.7	…..	2.11	2.8	2.42	2.56	…..	2.12	1.85
17	GNG-22 (RBD)	14.21	2.98	…..	…..	3.05	0.32	…..	…..	0.77	…..
18	GNG 4(RBD)	15.29	2.72	0.22	0.32	3.43	0.28	0.43	0.48	0.86	…..

*Deodorized samples (GNG-12-deodorized 3 times, GNG-13-deodorized 1 time)

****Refined samples (chemically refined samples-degummed, neutralized, bleached, winterized and deodorized)

Table 4: Mean and Standard Deviation of some fatty acids in unrefined camelina oils

Sample	C16:0	C18:0	C 18:1	C18:2	C18:3	C20:1	C22:1
	Palmitic	Stearic	Oleic	Linoleic	Linolenic	Eicosenoic	Erucic
Group-I							
GNG-1	7.27	3.9	15	15.55	22.85	13.36	1.56
GNG-3	6.72	3.38	19.30	21.04	23.9	14.26	0.37
GNG-5	6.47	3.27	20.12	19.91	24.54	15.04	0.18
GNG-8	5.61	3.65	15.66	17.15	24.13	12.86	2.33
GNG-9	6.37	3.88	18.67	21.58	24.3	14.16	2.92
GNG-10	6.56	3.42	18.82	21.83	25.04	14.08	2.93
GNG-14	6.95	3.37	19.3	24.31	23.5	12.79	0.29
Mean	6.56	3.55	18.12	20.20	24.04	13.79	1.51
SD	0.52	0.26	1.97	2.98	0.71	0.82	1.24
Group- II							
GNG-11	9.50	3.51	18.99	22.19	20.3	13.55	2.86
GNG-17	7.67	4.08	13.8	14.82	22.03	9.91	2.9
GNG-18	14.12	4.62	13.81	17.03	18.8	9.7	3.58
GNG 19	9.4	4.58	10.08	10.14	16.28	12.8	5.3
GNG 21	5.54	4.34	11.35	11.81	16.65	12.58	5.1
Mean	9.2	4.2	13.6	15.2	18.8	11.7	3.9
SD	3.2	0.5	3.4	4.7	2.4	1.8	1.2
p-value	0.131	0.025*	0.038*	0.080	0.007*	0.056	0.007*

*-significant difference at 95% confidence levels when p<=0.05, no significant difference when p >0.05

Table 4 shows the means and standard deviations determined for some major fatty acids found in unrefined camelina oil samples. In unrefined camelina oils, samples # 1, 3, 5, 8, 9, 10 and 14wax had very similar fatty acid compositions and suggest that these oil samples could be produced from very similar cultivars, growing location and conditions. Based on this observation, we had grouped it as group I set of samples and the samples which were not similar to this pattern as group II. The other unrefined oil samples set, Group II # GNG-11, 17, 18, 19 and 21, showed some variations. Sample # GNG-11 had a higher palmitic acid and lower linolenic acid content. Sample 18 had higher levels of palmitic and stearic acids and lower levels of oleic, linoleic, α-linolenic and eicosenoic acids. Sample GNG-17 was also low in oleic, linoleic and eicosenoic

acids, but comparable to the rest of samples (Table 3) in terms of palmitic, stearic and erucic acid levels (Table 4). Sample 19 and 21 had the highest levels of erucic acid in this study at 5.09 and 4.95% respectively. Samples GNG-19 was also high in palmitic acid and low in α-linolenic acid content.The variations in the major fatty acids observed in samples GNG-11, 17, 18, 19 and 21 could be due to cultivar variety and growing conditions (location and weather patterns) since the origin of these samples is not known. The levels of erucic acid from this study were very wide and some of them were higher than the level recommended by FDA. A student's T- test (with 2 tailed distribution at the 95% confidence level, where p-value or probability value equals 0.05) was performed using Microsoft excel between the 2 groups (Group I & II) of unrefined oil samples and highest significant difference was observed for α-linolenic ($p=0.007$), erucic ($p=0.009$), eicosenoic ($p=0.05$), oleic ($p=0.04$) and stearic acids ($p=0.021$). As seen from table 4,since p values of stearic, oleic ,α-linolenic, erucic, eicosenoic values were below $p=0.05$,this suggests that was significant difference in the levels of these fatty acids between the 2 groups of unrefined oil samples and no significant difference was observed for palmitic and linoleic acids. FDA recommends a max limit of 5 % of erucic acid in vegetable oils and erucic acid from cultivars used in production of unrefined oils # GNG 19 and 21 should be improved to meet this recommendation in edible oils.

The fatty acid composition in camelina oils characterized by this study follows a pattern which is comparable to the results reported previously (Budin et al.,1995, Putnam et al.,1993, Zubr and Matthaus,2002,Vollmann et al.,2007,Eidhin et al.,2003) . There were major variations in the levels of the major fatty acids in unrefined camelina oils and this probably could have been due to growing locations and conditions

and cultivar variety. Differences in the levels of major fatty acids in deodorized and refined oils were less and this could probably due to a very small number of samples being analyzed. Variations in the levels of the five major fatty acids in camelina oil due to different cultivars, regions and growing conditions have also been reported in the U.S and Europe by previous studies (Zubr and Matthaus, 2002, Angelini et al., 1997, H.Abramovic and V.Abram., 2005, Zubr, 2003). The α-linolenic acid levels from our study were lower than some of the ALA values 30.7 % (Budin et al., 1995) and 30.2% (Putnam et al., 1993) reported in camelina oil produced in the United States.

5.2 Unsaponifiables Fraction

5.2.1 Calculation and determination of Unsaponifiables fraction

The percent of unsaponifiables fraction was calculated using the following formula.

% Unsaponifiables matter = 100 × ((gm residue –gm blank residue) ÷gm sample).

The unsaponifiables fraction results are tabulated in Table 5 and it can be observed that the unsaponifiables fraction in unrefined and deodorized samples was found in the range 0.50-0.80%. In refined oil samples, the range was much smaller and found between 0.58 to 0.60%. In a study on the plant sterols in 14 commercial edible oils, the level of sterols in refined camelina oil was 0.55% (Schwartz et al, 2008). Refined vegetable oils contain 0.5-1.0% of unsaponifiables matter and the value reported in olive oil is 0.41%, soybean oil is 0.25% and corn germ oil is 0.97%(Monreau, 2006). Most fats and oils contain unsaponifiable compounds in the range between 0.2 to 1.5 % (Belitz and Grosch, 1999) and we observed that both the refined and unrefined oil samples in our study was within this range and similar to rapeseed (0.7-1.1%) and soya (0.6-1.2%).

The unsaponifiables fractions in refined samples were lower than that of unrefined oil samples and this reduction in unsaponifiables level could be due to the refining process. During refining of vegetable oils, a reduction in free sterol content and increase in esterified sterol fraction has been reported(Verleyen et al., 2002). Also, the content of tocopherols can be reduced, as much as 60% during the deodorization process of refining oils (Hernandez, 2004).

Table 5: Percentages of total unsaponifiables matter present in camelina oil samples

S.No	Sample description	Sample weight (in gm)	Weight of residue (in gm)	%Unsaponifiables matter
	UNREFINED			
1	GNG 19 Unrefined oil	0.2825	0.0013	0.46
2	GNG 18 Unrefined oil	0.2523	0.0017	0.67
3	GNG 1 Unrefined oil	0.266	0.002	0.75
4	GNG 17 Unrefined oil	0.2666	0.002	0.75
	Mean			0.66
	Standard Deviation			0.14
	DEODORIZED*			
5	GNG-13,Deodorized Camelina	0.2426	0.0019	0.78
6	GNG-12 Deodorized Camelina	0.2766	0.0013	0.47
	Mean			0.63
	Standard Deviation			0.22
	REFINED**			
7	GNG-4,Camelina Oil RBD	0.2404	0.0014	0.58
8	GNG 22 Camelina oil RBD	0.2984	0.0018	0.60
	Mean			0.59
	Standard Deviation			0.01

*Deodorized samples (GNG-12-deodorized 3 times, GNG-13-deodorized 1 time)

**Refined samples (chemically refined samples-degummed, neutralized, bleached, winterized and deodorized)

Table 5 also shows the means and standard deviations of unsaponifiables fraction determined in camelina oil samples. The unsaponifiables content in refined camelina oils GNG 4 and 22 were comparable to published reports (Shukla et al., 2002 and Schwartz, et al., 2008).

In the unrefined camelina oil samples, residue levels in GNG 18, GNG 17, and GNG 1 were very similar except one sample GNG 19(0.46%). The unsaponifiables level in sample GNG 13 (deodorized 1 time) was similar to unrefined oil samples GNG-1 but sample GNG 12 (deodorized 3 times) had a lower unsaponifiable residue level (0.47%). This could be due to the distillation of free sterols during the deodorization process and about 2 to 20% of phytosterols can be present in the deodorizer distillate obtained during the deodorization process (Verleyen et al., 2002).

5.2.2 Identification of Unsaponifiables components by GC-MS

A total of 21 different compounds were identified in the unsaponifiables fraction of 8 camelina oil samples. The unsaponifiables extract was composed of free sterols, tocopherols, diterpenes, triterpenes and hydrocarbons on identification. The major sterols found in camelina oil were β-sitosterol (31-46%), campesterol (8-30%), cholesterol (5.8-9.3%),brassicasterol (4.6-7.5%), γ-sitosterol (2.05-27%), δ^5-avenosterol (traces-11.75%) and phytol (2-9%). 14-Methylergost-8-en-3-ol(2-6%), stigmasterol (1.20-3.70%),obtusifoliol(1.70-6.0%) and cycloartenol(1.2-3.5%) were identified in most samples . Campesterol, stigmasterol and sitosterol are generally found in high levels in plant oils and are structurally very similar to cholesterol except the side chain on C-17. Δ5-Avenosterol is a derivative of sitosterol and obtusifoliol has also been identified in plant oils (Belitz & Grosch, 1999). Campesterol and stigamasterol concentrations in our study were comparable to the levels reported by Shukla et al., (2002).

22-Dehydrocholesterol and α1-sitosterol (citrostadienol) were found in most samples in small quantities (1-3%). 22-dehydrocholesterol has been identified in the sterol fraction of *Brassica napus* seed oil (Itoh et al., 2006). 6-Methyl cholest-5-en-3-ol was found in most of the samples in small quantities (0.1-0.9 %). Stellasterol was found in all samples in trace quantities and 24-desmethylsterol was identified in trace amount in only 1 sample # GNG 17. 14-methyl fecosterol was found high (9%) in 1 deodorized sample # GNG-13 and in some samples in trace levels. The GC-MS chromatogram of camelina oil unsaponifiables in sample #GNG-4 with peak assignments is shown in Figure 11.

Beta and γ- tocopherols (0.05-0.2%) were found in all samples and δ-tocopherol was identified in very small quantities (<0.2%) in samples GNG 4 and GNG 13. Vitamin E was found in most samples in small levels. Gamma-tocopherol is the main tocopherol in camelina oil and reported upto 72 mg/100 g oil (Schwartz et al., 2008).

Squalene was found in all samples in the range between 2.4-10.45%.Squalene is the biochemical precursor to the entire family of steroids and is a linear triterpene (Belitz and Grosch, 1999). It is a major hydrocarbon constituent of olive oil (1-7 g/kg of oil) and is used as a natural moisturizer in many skin care products (Kraft and Lynde, 2005). Squalane was found in small quantities (traces-1.0%) in 5 samples (GNG 1, GNG12, GNG 13, GNG4 and GNG 22). Squalane is a saturated form of squalene and often used in personal care products since it is less susceptible to oxidation than squalene. β–Amyrin, a triterpene alcohol was found in 6 samples in small quantities and the highest level was observed in refined oil sample # GNG 22(4.14%). Phytol was found in moderate levels in all camelina oil samples and this compound, is an acyclic diterpene alcohol usually esterified to chlorophyll in plants and. Phytol and isophytol are considered decomposition products of chlorophyll and squalene (Pudelkiewicz et al, 1983) and phytol was found highest in deodorized camelina samples in this study (Table 6).

Tretinoin (retinoic acid) is an oxidation product of Vitamin A and was found in 2 samples GNG 4 (2.60%) and GNG19 (0.70%). This compound has been reported to be used in acne treatment and also used in topical products intended for treatment of photoaging (Stefanaki et al., 2005). Sample GNG 12 contained trace levels of 1, 2-dihydrocurcumin.

Phytol, squalene, obtusifoliol 22-dehydrocholesterol, 6-methylcholest-5-en-3-ol, stellasterol, 14-methylergost-8-en-3-ol, 14-methylfecosterol, γ-sitosterol and β-amyrin were not identified in camelina oil by previous studies and we believe they are first being reported here. Table 6 summarizes the unsaponifiable compounds identified in this study and are expressed as the percentage of total unsaponifiables compounds. Table 7 gives the distribution of unsaponifiable compounds in camelina oil based on total weight of unsaponifiables (mg/100 g oil). The mass spectra of some of the unsaponifiable compounds found in this study are illustrated in Figure 12.

Major unsaponifiable compounds in refined oils: Major sterols identified were β-sitosterol, campesterol, cholesterol, brassicasterol, 14-methylergost-8-en-3-ol, cycloartenol and obtusifoliol (Table 6 and 7). Phytol and Squalene were found in moderate levels and squalane, α, β, and γ-tocopherols were found in both the samples. Vitamin E was found in trace amounts in both the refined oil samples and retinoic acid was found in sample GNG-4 at a high level when compared to other camelina oil samples in this study (Table 6).

Major unsaponifiable compounds in deodorized oils: The major sterols were γ-sitosterol, campesterol, δ^5-avenosterol, cholesterol, brassicasterol, 14-methylfecosterol, obtusifoliol and β-sitosterol. Δ5-Avenosterol has been reported in virgin olive oil and has also been suggested to contribute to anti-oxidant stability of virgin olive oil (Moreau, 2004). Small levels of 14-methylergost-8-en-3-ol, stigmasterol, cycloartenol and 22-dehydro-cholesterol were also observed (Table 6). Phytol, γ-sitosterol and squalene were high in these samples when compared to unrefined and refined oil samples. The presence

of γ-sitosterol in deodorized oil unsaponifiable fraction could possibly be due to the conversion of the beta- isomer to the gamma- form during the deodorization process. Also α-, β- and γ-tocopherols were found in small quantities and both samples contained trace amounts of stellasterol.

Major unsaponifiable compounds in unrefined oils: β-sitosterol, campesterol, cholesterol and brassicasterol were the major compounds in these samples (Tables 6 and 7). 22-dehydrocholesterol, 14-methylergost-8-en-3-ol, stigmasterol, obtusifoliol, cycoartenol, α-1sitosterol, β-amyrin and Vitamin E were found in small quantities. Phytol and squalene were found in moderate amounts and the levels of squalene were lower than deodorized samples. Squalane was found in only 1 sample GNG 1.

The concentration range of some of the unsaponifiable compounds in unrefined, deodorized and refined camelina oil samples are summarized in Table 8. The sterol values reported in the study by Shukla et.al (2002) were re-calculated in this study to express it in mg/100 g oil. Cholesterol levels were high in unrefined camelina oil samples.γ-sitosterol and δ^5-avenosterol were found in high levels in deodorized samples and refined oil samples had the highest level of cycloartenol and β-amyrin in this study. It can also be seen that average cholesterol and brassicasterol values in our study were much higher than published reports on camelina sterol fraction.

The unsaponifiable fraction identified in camelina oil follows the pattern reported previously in camelina oil. The unsaponifiable fraction of camelina makes it suitable for use in food, nutraceutical and pharmaceutical products.

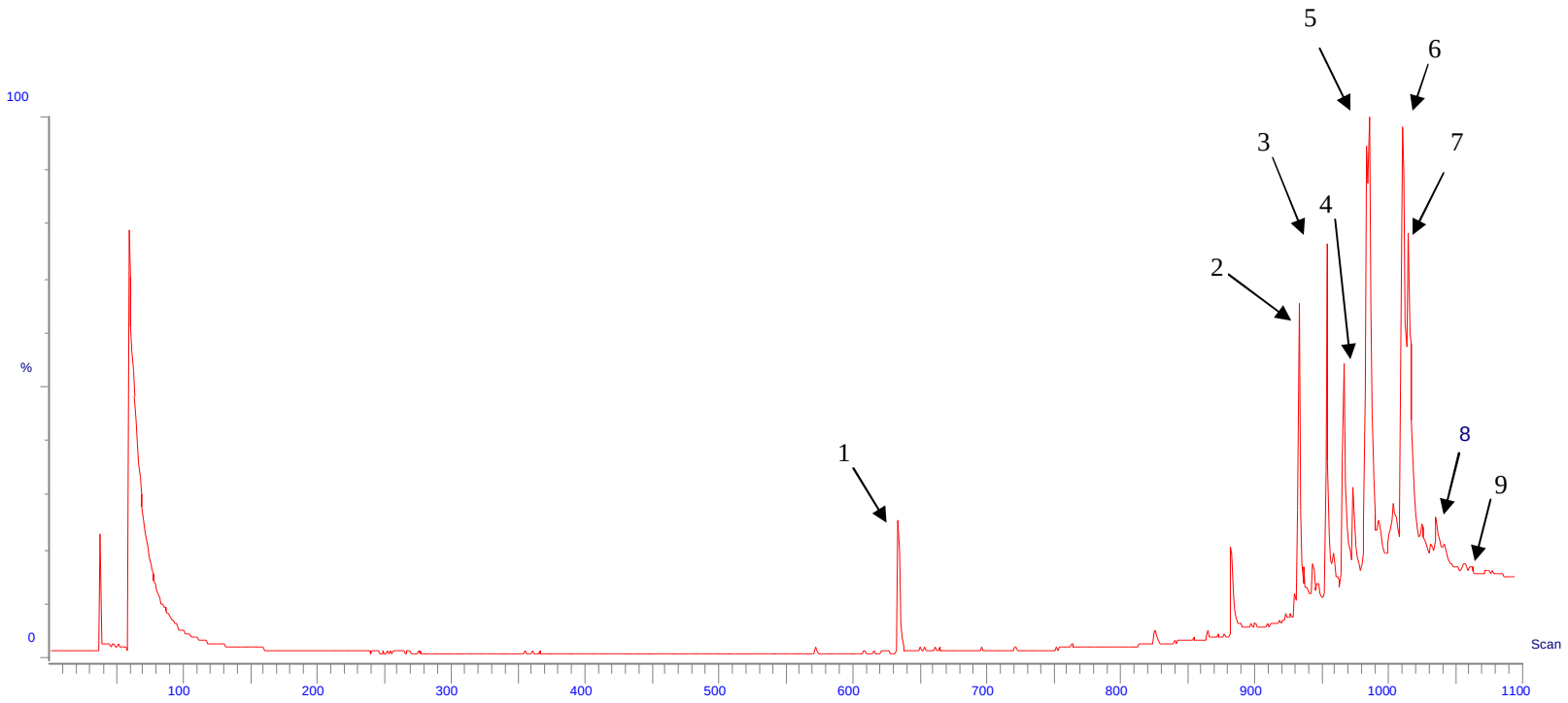


Figure 11: GC-MS Chromatogram of Camelina Oil Unsaponifiables fraction of sample # GNG-4

1. Phytol 2. Squalene 3. Cholesterol 4. Brassicasterol 5. Campesterol + Stigmasterol 6. β-Sitosterol 7.δ^5-Avenosterol 8. Cycloartenol 9. β-Amyrin.

Table 6: Unsaponifiables Compounds identified and their percentage distribution in 8 Camelina Oil Samples.

Compound	GNG17	GNG-19	GNG1	GNG18	GNG-12	GNG 13	GNG-4	GNG-22
	Unrefined	Unrefined	Unrefined	Unrefined	Deodorized*	Deodorized*	Refined**	Refined**
phytol	6.4	1.97	2.39	5.89	8.99	8.92	2.34	7.56
B & γ- tocopherol	0.2	0.2	0.15	0.1	0.15	0.15	0.2	0.05
squalene	8.2	2.4	5.7	3.51	10.45	8.2	7.28	5.22
δ- tocopherol	…..	….	…..	…..	traces	…..	0.19	…..
22-dehydrocholesterol	2.6	traces	1.8	0.64	2.98	2.07	…..	…..
retinoic acid	…..	0.67	…..	…	…..	…..	2.58	…..
cholesterol	9	7.9	8.4	7.77	7.55	9.5	9.3	5.78
6-methylcholest-5-en-3-ol	0.24	0.1	0.07	0.85	…..	0.2	…..	…..
brassicasterol	6.36	5.45	5.81	4.99	7.49	6.47	6.8	4.62
14-methylergost-8-en-3-ol	4.7	1.94	3.62	3.71	6.02	5.51	3.87	4.16
14-methylfecosterol	……	traces	traces	….	9.06	…..	…..	…..
campesterol	21.53	27.48	24.9	29.63	7.58	20.41	25.52	19.75
stigmasterol	2.82	1.14		traces	3.65	3.15	traces	traces
obtusifoliol	4.83	1.73	4.74	2.25	5.6	5.71	6.02	…..
β-sitosterol	29.04	45.6	36.35	39.87	8.3	traces	31.4	37.16
γ-sitosterol	traces	2.05	traces	traces	traces	27.38	…..	…..
δ5-avenosterol	traces	traces	traces	traces	11.75	traces	traces	…..
cycloartenol	2.7	1.23	2.79	0.79	3.52	2.37	3.87	10.84
Vitamin E	1.43	….	traces	…	3.26	traces	traces	traces
β-amyrin	…..	traces	1.96	…	1.3	…..	traces	4.14
α-1-sitosterol	traces	0.1544	1.04	traces	…..	…..	traces	…..

* Deodorized samples (GNG-12-deodorized 3 times, GNG-13-deodorized 1 time)

******Refined samples (chemically refined samples-degummed, neutralized, bleached, winterized and deodorized)

Table 7: Unsaponifiables compounds distribution based on total wt of unsaponifiables (mg/100 g oil) in 8 camelina oil samples

Compound	GNG17	GNG-19	GNG1	GNG18	GNG-12	GNG 13	GNG-4	GNG-22
	Unrefined	Unrefined	Unrefined	Unrefined	Deodorized*	Deodorized*	Refined**	Refined**
Unsaponifiables	**750**	**460**	**750**	**670**	**470**	**780**	**580**	**600**
phytol	47.9	9.1	17.9	39.4	42.2	70	15.4	45.4
squalene	63	11.8	44	24	49.83	65.2	41.9	31.32
δ-tocopherol	…..	….	…..	…..	traces	…..	1.1	…..
22-dehydrocholesterol	19.3	traces	13.4	4.3	traces	16.1	…..	…..
retinoic acid	…..	3	…..	…	…..	…..	14.9	…..
cholesterol	69.2	36.7	63.5	52.1	35.4	75.5	53.8	34.00
6-methylcholest-5-en-3-ol	traces	traces	traces	6.00	…..	traces	…..	…..
brassicasterol	47.7	25.1	43.6	33.5	35.2	50.5	39.4	27.7
14-methylergost-8-en-3-ol	35.2	8.9	27.1	25	28.2	43.0	22.5	24.9
14-methylfecosterol	……	traces	traces	….	42.6	…..	…..	…..
campesterol	161	126	187	198	35.6	159	148	118.5
stigmasterol	21.1	5.2	…..	traces	17.1	24.4	traces	traces
obtusifoliol	36.2	8	35.6	15.1	26.3	44.5	34.9	…..
β-sitosterol	218	219	273	267	39	….	182	223
γ -sitosterol	traces	traces	traces	traces	traces	213	…..	…..
δ5-avenosterol	traces	traces	traces	traces	55.1	traces	traces	…..
cycloartenol	20.1	5.6	21.00	5.3	16.5	18.5	22.4	65
Vitamin E	10.7	….	traces	…	5.7	traces	traces	traces
β -amyrin	…..	traces	15.00	…	15.8	…..	traces	24.8
α-1-sitosterol	traces	0.7	8.00	traces	…..	…..	traces	…..

***** Deodorized samples (GNG-12-deodorized 3 times, GNG-13-deodorized 1 time)

******Refined samples (chemically refined samples-degummed, neutralized, bleached, winterized and deodorized)

Table 8: Unsaponifiables compounds (mg/100 g oil) in camelina oil common to our study and published data.

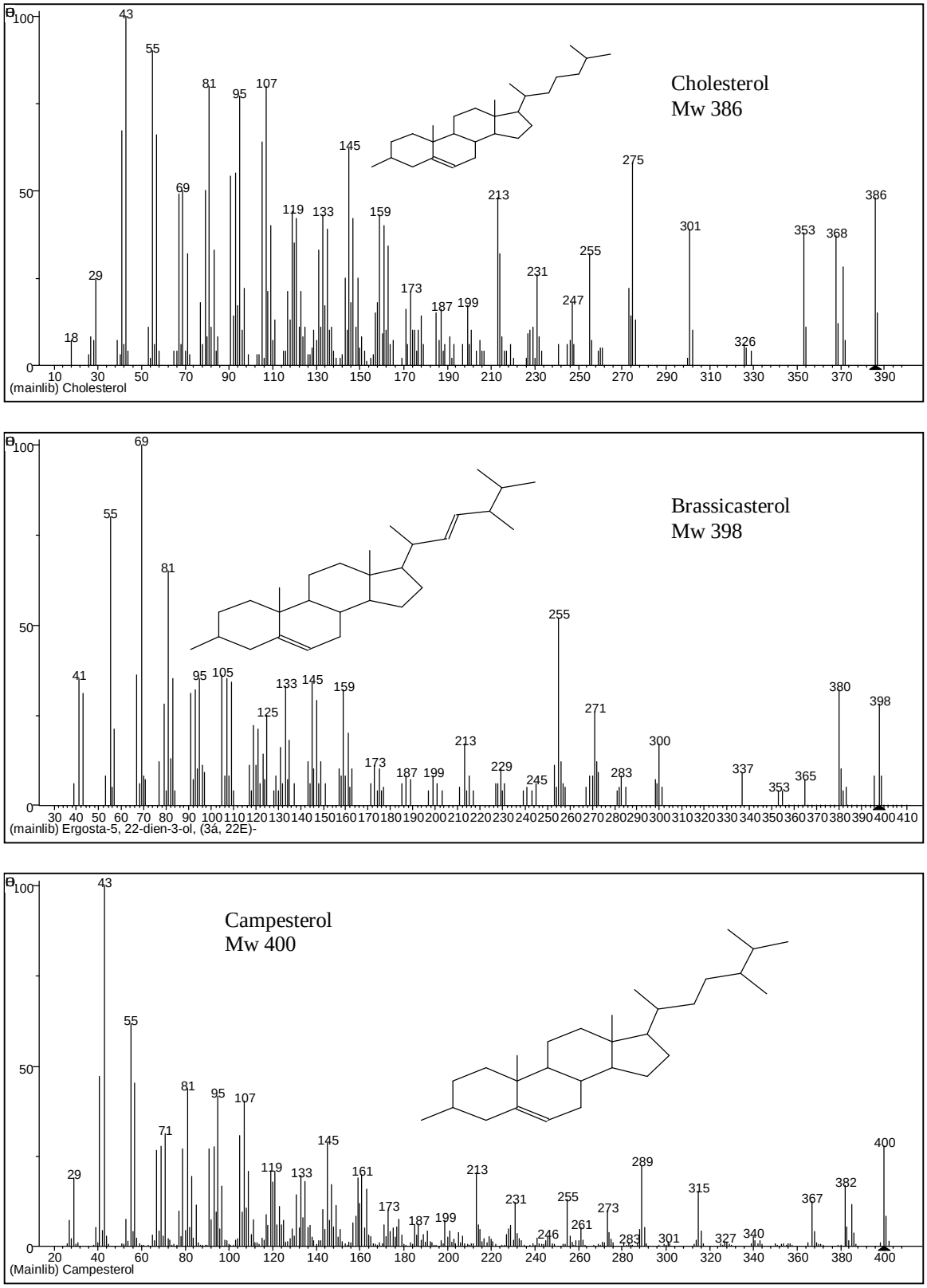
Compound	Unrefined	Deodorized*	Refined**	Schwartz et al.,2008	Shukla et al 2002
phytol	9.0-48.0	42.0-70.0	15.5-45.0	….	….
squalene	12.0-63.0	49.0-65.0	31.0-42.0	….	…..
22-dehydrocholesterol	0.0-19.3	0.0-16.1	….	….	….
cholesterol	36.7-69.0	35.0-75.0	34.0-54.0	35	18.8
brassicasterol	25.0-48.0	35.0-51.0	28.0-40.0	27	13.3
14-methylergost-8-en-3-ol	9.0-35.0	29.0-43.0	23.0-25.0	….	…..
campesterol	126-198	35.6-159	119-148	117	89.3
campestanol	…..	…..	…..	1.6	….
stigmasterol	5.0-21.0	17.0-25.0	….	5.6	10.3
obtusifoliol	8.0-36.0	27.0-45.0	0.0-35.0	…..	…..
β-sitosterol	218-273	0.0-39.0	183-223	300	188.4
γ -sitosterol	traces	0.0-213	….	…..	…..
α-1-sitosterol	0.7-8.0	….	…..	1.3	…..
sitostanol	…..	…..	…..	2.5	…..
δ5-avenosterol	traces	0.0-55.0	traces	37	39.3
cycloartenol	5.0-21.0	16.5-18.5	22.5-65	10	51.5
Vitamin E	traces-10.7	0-5.7	traces	…..	…..
β-amyrin	traces-15	0-15.8	0-24.8	…..	…..
24-methylene cycloartenol	…..	…..	…..	1	12.4
Others	5.85	39.18	12.54	8	116.7
Total Unsaponifiables	**657+/-137**	**630+/-220**	**590+/-14**	**546**	**540.4**

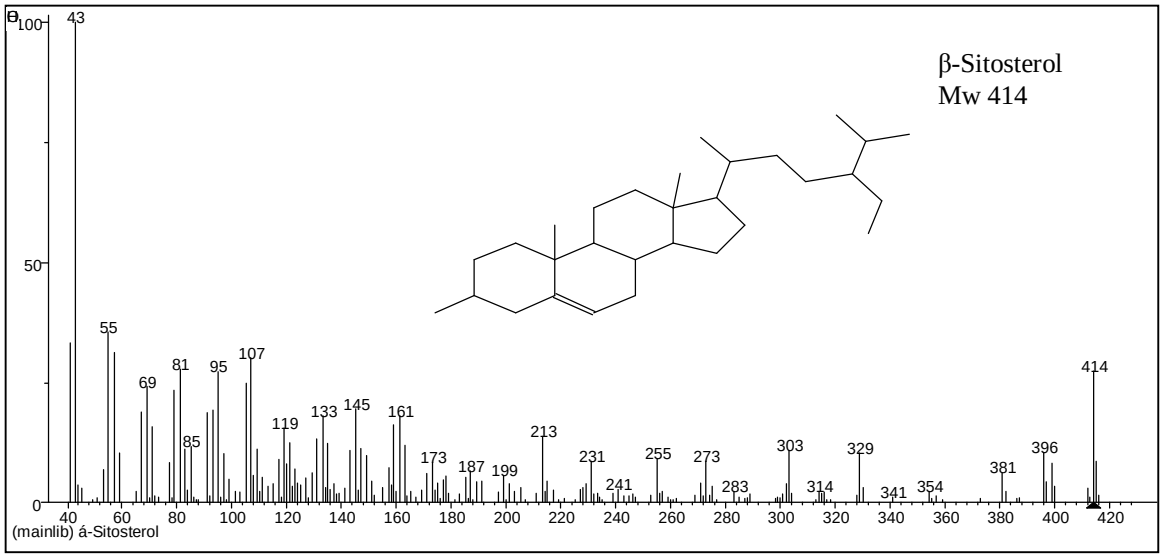
* Deodorized samples (GNG-12-deodorized 3 times, GNG-13-deodorized 1 time)

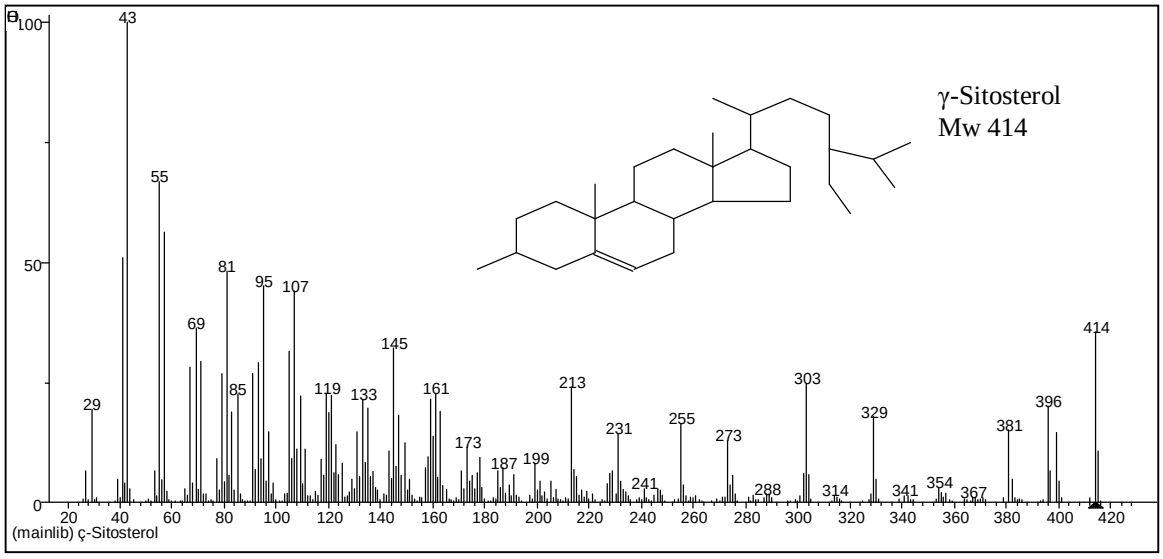
******Refined samples (chemically refined samples-degummed, neutralized, bleached, winterized and deodorized)

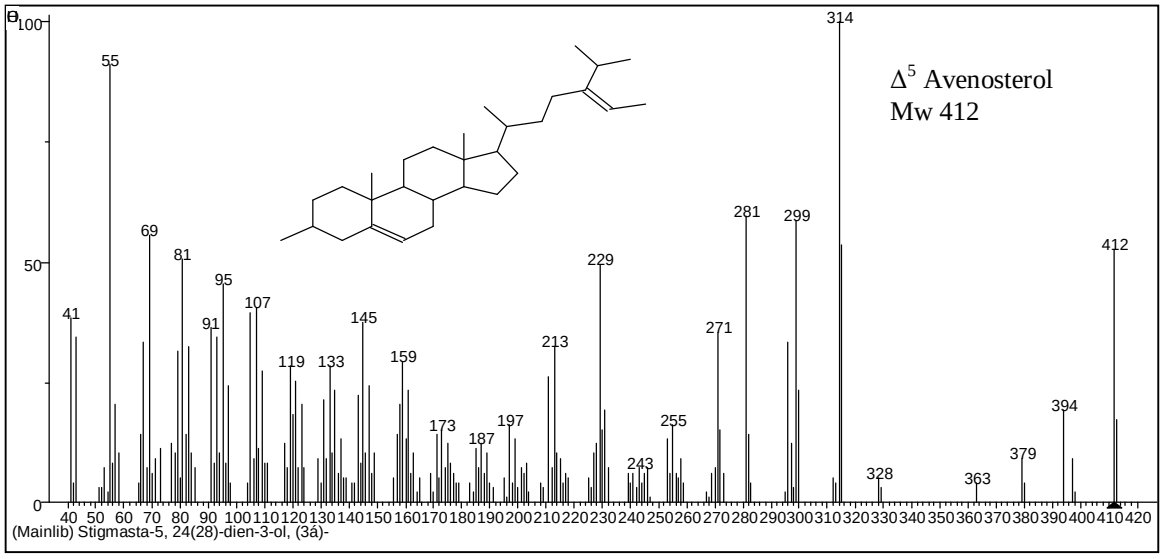
Table 8 compares the concentration range of unsaponifiables compounds identified in this study and published data. The sterol composition of vegetable oils can be influenced by genetic species, growing and storage conditions, influence of the refining process and specificity of the analytical method applied (Verleyen et al., 2002) and we can see huge variations in the sterol compositions from this study (Table 8). Cholesterol, brassicasterol and campesterol were high in this study when compared to other reports. Beta-sitosterol levels in unrefined and refined oils were comparable to the level reported by Shukla et al., (2002) but lower than level reported by Schwartz et al., (2008). Samples # GNG 18 and 19 were the best sources of β-sitosterol and campesterol.

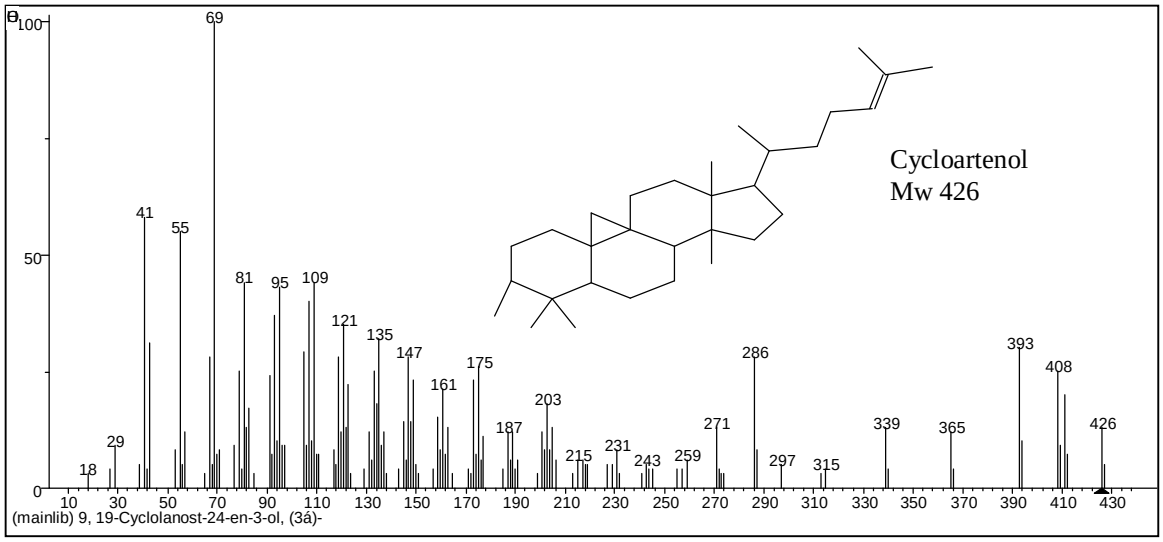
Figure 12: Mass-spectra of unsaponifiables compounds present in camelina oil.

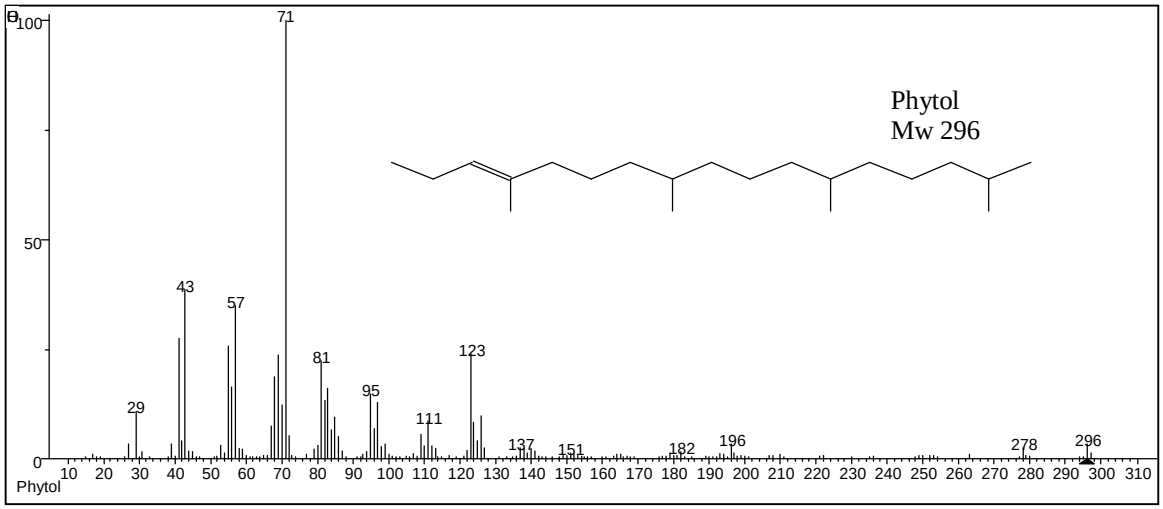


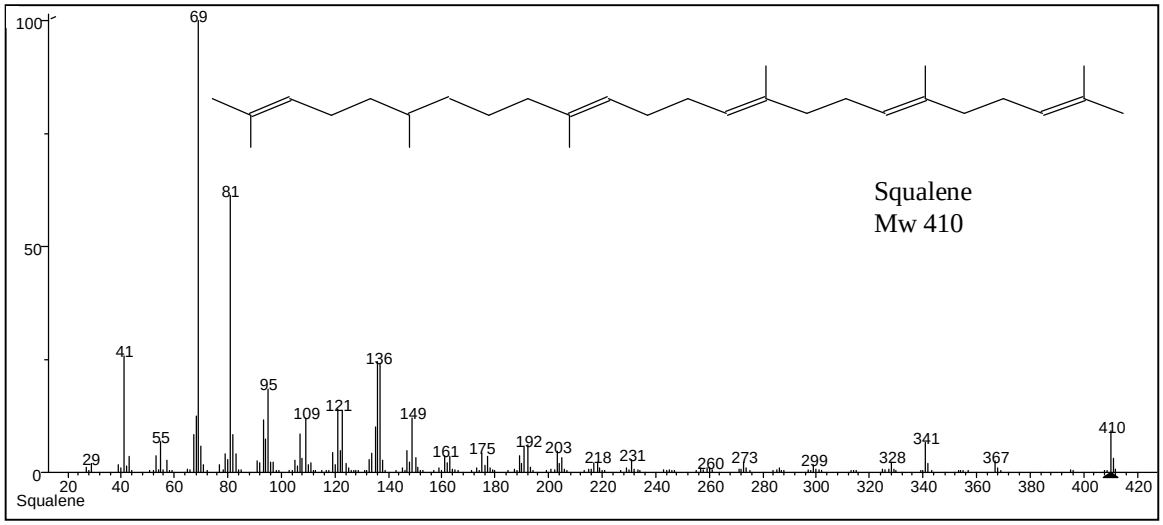

β-Sitosterol
Mw 414
(mainlib) á-Sitosterol


γ-Sitosterol
Mw 414
(mainlib) ç-Sitosterol


Δ5 Avenosterol
Mw 412
(Mainlib) Stigmasta-5, 24(28)-dien-3-ol, (3á)-


Cycloartenol
Mw 426
(mainlib) 9, 19-Cyclolanost-24-en-3-ol, (3á)-


Phytol
Mw 296
Phytol


Squalene
Mw 410
Squalene

5.3. FREE FATTY ACIDS

Table 9 shows the values of free fatty acids determined in unrefined, deodorized and refined camelina oil samples.

Table 9: Free Fatty acids determined in camelina oil samples.

S.No	Sample description	%FFA
		as oleic acid
	UNREFINED	
1	GNG 19, Unrefined oil	0.65
2	GNG 18, Unrefined oil	0.50
3	GNG 1,Unrefined oil	0.80
4	GNG 17 ,Unrefined oil	0.55
	Mean	0.625
	SD	0.13
	DEODORIZED*	
5	GNG-13,Deodorized Camelina	0.65
6	GNG-12 Deodorized Camelina	0.70
	Mean	0.675
	SD	0.035
	REFINED**	
7	GNG-4,Camelina Oil RBD	0.03
8	GNG 22 Camelina oil RBD	0.06
	Mean	0.045
	SD	0.02

* Deodorized samples (GNG-12-deodorized 3 times, GNG-13-deodorized 1 time)

**Refined samples (chemically refined samples-degummed, neutralized, bleached, winterized and deodorized)

Free fatty acids (FFA) were present in the range between 0.55 to 0.80 % in unrefined camelina oil samples. This parameter represents the content of free fatty acids present in unrefined camelina oil and is a measure of the treatment of seeds before and during pressing. The FFA levels in the deodorized samples were lower than the level of FFA in original unrefined sample # GNG-1. Abramovic et al., (2007) reported FFA as 0.54% in fresh camelina oil and this increased with storage time up to 0.74% after 44 days. A FFA value of 2.35% was reported in fresh camelina oil produced at Prevalje, in Korosk'a region, Slovenia (H.Abramovic and V.Abram, 2005). In all cases, the FFA

determined in this study was lower than the recommended level (less than 1%) in unrefined edible oils.

FFA levels in refined oil samples were low (0.03-0.06%) and this is typical since the first step in refining oils is neutralizing the oils which helps in removing the free fatty acids. The FFA levels in refined camelina oil samples were below the preferred ideal limit (less than 0.1%) in refined vegetable oils (Hernandez, 2004). Reduction in free fatty acid values from 2.3% in crude soybean and corn oils to 0.08-0.11% and 0.08% by physical and chemical refining processes respectively were reported (Verleyen et al., 2002).

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5.4 Volatiles and Semi-volatiles

5.4.1 Identification of volatiles and semi-volatile compounds in Camelina seeds

Camelina seeds belong to the cruciferous seeds family which is considered aromatic but since camelina seeds fell into disfavor due to its glucosinolate and erucic acid content, not much work has been done on its flavor and aroma profile. Camelina seeds have been described as having a bitter and cabbage trunk or kohlrabi taste (Mikersch et al., 1952). In a study done to evaluate the volatile compounds found in the headspace of soybean seeds at various developmental stages, trans-2-heptenal, trans-2-octenal, ethanol, 1-hexanol, and 1-octen-3-ol were identified in high levels at seed maturity stage (Boue et al., 2003).

A total of 168 compounds were identified in the headspace volatiles of camelina seeds and are summarized in Table 10. Acids, alcohols, esters, carbonyl compounds (ketones and aldehydes), hydrocarbons (alkanes, alkenes, and alkynes), aromatic hydrocarbons, ethers, pyrazines, terpenes and sulfur containing compounds were identified. Retention indices for these compounds were determined based on n-alkanes hydrocarbon from C-5 to C-18 present in camelina seed samples. Concentration of sample volatile compounds was calculated by dividing the integrated peak area of the flavor components by internal standard (toluene-d_8) using GC-MS data.

Three fatty acids namely acetic, hexanoic and nonanoic were identified in seeds and acetic acid was found in all samples.

A total of five (5) ether compounds were identified and 2-ethyl furan and 2-pentyl furan were identified in all samples. 2-Ethyl furan gives a sweet, burnt and earthy note and 2-pentyl furan is usually associated with buttery, green-bean like aroma. 2-Pentyl

furan is an oxidation product of linoleic acid (C18:2, n-6) which is responsible for the flavor reversion phenomenon in soybean oil (Ho and Chen, 1994). 2-Pentylfuran and 2-ethyl furan were found in remarkable amounts in linseed oil samples from seeds that were pre-heated (60° C for 30 minutes) when compared to seeds which were not pre-heated (Krist et al., 2006).

Hydrocarbons (alkanes-50 and alkenes-18) were identified in the seed samples. The hydrocarbons do not have a great impact on the aroma and flavor profile because of their high detection threshold (90-2150 ppm) (Frankel, 1997). About 14 aromatic benzene compounds like toluene, naphthalene, xylene, cymene and styrene were identified in most of the camelina seed samples.

Phenolic compounds (3) were identified and these compounds are responsible for a smoky, woody and phenolic note.

A total of 30 alcohol compounds were identified and the most common being ethanol, isopropyl alcohol, isoamyl alcohol, isobutyl alcohol and 2-methyl-1-butanol. Compounds like 1-butanethiol, eucalyptol, heptanol, cis-3-hexenol and 1-nonanol have odor detection thresholds between 3-50 ppb and can exert an influence on the aroma profile of camelina seeds.

A total of 26 aldehyde compounds were identified in camelina seeds, which consisted of saturated and unsaturated compounds. Aldehydes, 2-alkenals and 2, 4-alkadienals are compounds arising from the lipid oxidation of the mono- and poly-unsaturated fatty acids. Aldehydes like heptanal, octanal, nonanal, decanal, 2-alkenals like 2-nonenal, 2-decenal and 2-undecenal arise from oleic acid oxidation. Acetaldehyde, pentanal, hexanal, 2-heptenal, 2-octenal, 2-nonenal, 2, 4-nonadienal and 2, 4-decadienal

are the main compounds arising from linoleic acid oxidation. Similarly propanal, butanal,acetaldehyde,2-butenal,2-pentenal,2-/3-hexenal,2,4-heptadienal and 3,6-nonadienal are the aldehyde compounds arising from the auto-oxidation of linolenic acid(Frankel 1997). The lipid oxidation products listed above were identified in camelina seed volatiles in varying concentrations.

A number of ketones (23) which are associated with a fruity note were identified in camelina seeds and these products arise due to decomposition of the secondary lipid oxidation products (Salinas et al., 1994). Most of the ketones except 3-boranone, carvone and acetophenone were present in all samples.

A total of 12 ester compounds were identified in camelina seeds. The ester methyl acetate found in most of the samples and the most of the esters were methyl esters of fatty acids.

Nitrogen-containing heterocyclic compounds (6) were identified in some camelina seed samples. 2-Sec-butyl-3-methoxy pyrazine is a powerful compound since it has a very low odor detection threshold of 0.001 μg/L water with an earthy, green-bell pepper, green pea kind of aroma (Belitz and Grosch, 1999). Similarly the pyrazine, 2-isopropyl-3-methoxy pyrazine is associated with a potato; mild nutty, musty and earthy kind of note and these pyrazines are believed to be metabolic by-products in some plant foods and microorganisms (Belitz and Grosch, 1999).

A total of 10 terpene compounds were identified and the compound limonene was found in all samples .This compound is associated with an orange-peel and citrus note. Cis-ocimene and β-myrcene are associated with a fresh and leafy green aroma. Compounds like α-pinene, β-pinene and α-thujene are associated with a fresh herbal and

spicy aroma. Sabinene, β-pinene and α-pinene have been identified in the essential oil of several spice plants (Belitz and Grosch, 1999). Monoterpenes like limonene, sabinene and β-myrcene were identified in the volatiles emitted by rapeseed (*Brassica napus* ssp. oleifera) during the growing season (McEwan and Macfarlane, 1998).

Sulfur containing compounds (14) were identified in camelina seed samples and they constitute the most powerful group in the headspace volatiles of camelina seeds as most of these compounds have very low odor detection thresholds. The compounds dimethyl sulfide (cooked cabbage, corn and seafood), dimethyl disulfide (cabbage, onion) and dimethyl trisulfide (cooked onion and meaty) were found in all samples and the detection thresholds of these compounds are 0.3-1.0ppm, 0.16-12ppm and 0.005-0.01 ppm respectively (Leffingwell, 1991). Methyl mercaptan and dimethyl disulfide are usually derived from methional, a non-enzymatic browning compound. Further decomposition of dimethyl disulfide yields dimethyl trisulfide and dimethyl tetra sulfide formed by disporportionation (Belitz and Grosh, 1999). Dimethyl disulfide, the respiratory irritant was also identified in the volatiles emitted by rapeseed during growing season (McEwan and Macfarlane, 1998). The compound allyl isothiocyanate was identified only in 1 sample # GNG-omega meal and this compound is obtained from sinigrin which is responsible for the mustard aroma in camelina meal. Carbonyl sulfide is naturally present in cheese, grains, seeds and certain vegetables (like cabbage) and is usually associated with an unpleasant sulfury odor. Compounds 2, 4, 5-trithiahexane and 2, 4-dithiapentane were also identified in some samples and are associated with cabbage and truffle like note respectively.

Table 10 summarizes the compounds identified in study of volatiles from camelina seed samples and Table 11 shows the number of volatile compounds identified in each camelina seed sample.

Table 10: Volatiles and semi-volatile compounds identified in camelina seeds.

Total compounds identified:168	
Acids(3 identified)	
acetic acid	hexanoic acid
nonanoic acid	
Alcohols(30 identified)	
ethanol	benzyl alcohol
isopropyl alcohol	phenyl ethyl alcohol
2-penten-1-ol	isoamyl alcohol
n-propanol	isobutyl alcohol
heptanol	1-butanol,2-butanol
1-octanol	5-methyl-1-hexanol
1-nonanol	thymol
tridecanol	1-decanol
2-penten-1-ol	2-heptanol
ethanol,2-butoxy	3-hepten-1-ol
1-heptyn-3-ol	1-butanol,2-methyl
3-nonen-1-ol	cis-3-hexen-1-ol
2-decen-1-ol	hexyl alcohol
tert-butyl alcohol	eucalyptol
5-hepten-2-ol,6-methyl	cyclohexanol,1-methyl-4-(1-methylethyl)
Esters(12 identified)	
methyl acetate	4-terpineol acetate
methyl hexanoate	ethyl palmitate
methyl octanoate	methyl linoleate
bornyl acetate	methyl oleate
methyl palmitate	phenyl acetate
p-menth-8-en-1-ol,acetate	
Phenolic compounds(3 identified)	
3,5-di-tert butyl phenol	2,6-di-tert butyl phenol
4-tert-octyl-o-cresol	
Carbonyl compounds-Aldehydes(26 identified)	
acetaldehyde	propanal,2-methyl
butanal,3-methyl	butanal,2-methyl
pentanal	hexanal
furfural	heptanal
benzaldehyde	octanal
nonanal	decanal
2-hepten-1-al	2-octenal
2-nonen-1-al	dodecanal
tetradecanal	tridecanal
phenyl acetaldehyde	2-pentenal
2-hexenal,3-hexenal	2,4-heptadienal(E,E)

undecanal	2,4-decadienal(E,E)
2,4-nonadienal(E,E)	2,4-hexadienal(E,E)
Carbonyl compounds-Ketones(23 identified)	
acetone	3-buten-2-one
2-butanone	acetoin
carvone	cycloheptanone,4-methyl
2-pentadecanone 6,10,14-trimethyl	5-hepten-2-one,6-methyl
3,5-octadien-2-one(E,E)	acetophenone
γ -decalactone	2-nonanone
2-pentanone	2-hexanone
2-heptanone	2-decanone
diacetyl	2-cctanone
3-boranone	3,5-heptadien-2-one,6-methyl
7-octen-2-one	2-undecanone
2-dodecanone	
Hydrocarbons-Alkanes(50 identified)	
pentane	hexane
undecane	2,2,5-trimethyl decane
tetradecane	2,3,5,8-tetramethyl decane
pentadecane	hexadecane
dodecane	tetradecane 2,6,10-trimethyl
tridecane	decane,2,2,3-trimethyl
2,5,6-trimethyl decane	2,6,7-trimethyl decane
4,6-dimethyl dodecane	2,2,5-trimethyl decane
tetradecane,3-methyl	2,6,6-trimethyl decane
tridecane,3-methyl	dodecane-2,6,11-trimethyl
heptadecane	tridecane,5-propyl
heptane	pentane,2,4-dimethyl
tridecane,2,5-dimethyl	tetradecane,2-methyl
dodecane,2-methyl-4-propyl	2,2,7,7-tetramethy octane
pentane,2-methyl	6-methyl tridecane
octane,2,6-dimethyl	pentadecane,7-methyl
decane,3-methyl	butane,2,2-dimethyl
decane,2,3,4-trimethyl	heptane,3-methyl
1-undecane,4-methyl	nonane,2-methyl
decane,3,4-dimethyl	nonane,4-methyl
undecane 2,9-dimethyl	nonane 2,5-dimethyl
dodecane,2,5-dimethyl	nonane,4,5-dimethyl
decane,6-ethyl,2-methyl	decane,3,6-dimethyl
undecane 3,9-dimethyl	undecane,3-methyl
tridecane,4,8-dimethyl	pentadecane,2-methyl
hexyl cyclohexane	
Hydrocarbon-Alkenes(19 identified)	
1-pentene,2-methyl	5-methyl-2-hexene

1-pentadecene	1-hexene
2-decene,6-methyl	5-tetradecene
7-tetradecene	2-dodecene
1-heptene,6-methyl	2-butene,2-methyl
1-tetradecene	1-octene,2-octene
1-pentene	1-tridecene
1-undecene	5-undecene
6-dodecene	4,4-dimethyl-1-hexene
diisoamylene	
Hydrocarbon-Aromatics(14 identified)	
2-methyl naphthalene	toluene
1-methyl naphthalene	o-xylene
1,3,5-trimethyl benzene	p-xylene
o-cymene	m-xylene
m-cymene	styrene
naphthalene-1,2,3,4,4A,5,8,8A-octahydro-4A-methyl(trans)	benzene,1,2-dimethoxy
p-cymene	p-dichlorobenzene
naphthalene	
Ethers (5 identified)	
2-methyl furan	2-ethyl furan
2-pentyl furan	furan,2,3-dihydro-4-methyl
2n-butyl furan	
N-containing heterocyclics(6 identified)	
2-methyl pyrazine	2,5-dimethyl pyrazine
2-methoxy-3-sec-butylpyrazine	2-isopropyl-3-methoxypyrazine
2-ethyl pyrazine	pyrazine,2-methyl-6-methylthio-
Terpenes (10 identified)	
limonene	α-tureen
ocimene, cis	β-pentene
3-carene	α-pinene
α-phellandrene	β-myrcene
sabinene	γ-terpinene
Sulfur compounds(14 identified)	
carbonyl sulfide	carbon disulfide
dimethyl sulfide	dimethyl disulfide
methyl mercaptan	dimethyl trisulfide
1-propanethiol	2,4,5-trithiahexane
1-butene,4-isothiocyanato	2,4-dithiapentane
propane,1-(methylthio)(same as methyl propyl sulfide)	ethanol,2-(methylthio)-
1-butanethiol	allyl iso thiocyanate

Table 11: Total number of compounds identified in individual camelina seed samples.

S.No	Sample Description	Type	Method	Total no of compounds
1	Ligena 1 seeds	Seeds	HDSP	26
			P&-T-TD	45
			Total	**71**
2	Ligena 1A seeds	Seeds	HDSP	24
			P&-T-TD	44
			Total	**68**
3	Calena 2 seeds	Seeds	HDSP	27
			P&-T-TD	38
			Total	**65**
4	Calena 2A seeds	Seeds	HDSP	24
			P&-T-TD	47
			Total	**71**
5	GNG-Omega meal	Meal	HDSP	27
			P&-T-TD	115
			Total	**142**
6	Ligena 1 Crushed	Crushed seeds	HDSP	24
			P&-T-TD	54
			Total	**78**
7	Ligena 1Preheated	Preheated & crushed	P&T-TD	**61**

It can be seen that the sensitivity of the purge-and-trap method complimented by the static headspace (HDSP) method has been helpful in determining the aroma profile of camelina seeds. Sample # Ligena 1 crushed seeds had a higher number of volatiles than sample # Ligena 1 seeds. Similarly Ligena 1 preheated & crushed sample had more number of volatiles identified than Ligena 1 seeds. The maximum number of volatile compounds was identified in GNG-Omega meal sample and this could affect the acceptability of the omega meal when incorporated in animal feed formulations. A number of sulfur compounds were identified in rapeseeds (*Brassica napus* ssp.oleifera) as a result of glucosinolate metabolism when the plant was damaged (McEwan and Macfarlane, 1998).

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5.4.2: Identification of volatiles and semi-volatiles in individual seed samples.

The volatiles and semi-volatiles compounds identified in individual camelina seed samples will be discussed in this section

5.4.2.1: Volatiles and semi-volatile compounds in Sample Ligena 1 seeds (by static and dynamic headspace volatiles method)

The volatiles and semi-volatile compounds (71) identified in sample Ligena 1 seeds are presented in Table 12a and 12b. The static headspace volatiles consisted of the lower molecular weight compounds and were dominated by sulfide compounds, aldehydes, alcohols and ketones. Acetaldehyde, 2-methyl butanal, 3-methyl butanal, pentanal, dimethyl sulfide, dimethyl disulfide, ethanol, isobutyl alcohol and isopropyl alcohol dominated the static headspace volatiles.

The dynamic headspace (purge-and-trap method) volatiles consisted of 2-alkenals, hydrocarbons, esters, terpenes and phenolic compounds. Major aldehydes were nonanal, heptanal, and hexanal, and alkenals identified were 2-nonenal, 2-octenal and 2-heptenal. Small levels of 2-pentyl furan, phenyl acetaldehyde, benzaldehyde, carvone, gamma-terpinene, dodecanal and 1-butene-4-isothicyanato were also identified in this sample. The compound 2-pentadecanone-6, 10, 15-trimethyl was identified in this sample and this compound has been identified in the essential oil of *Pinus caribaea* Morelet dry needles upto 2.70% and is responsible for mild fruity and brown note (Chowdhury et al.2008).

The aroma of the out-gas products from this sample (evaluated from the tenax trap end) can be described as sulfury (typical cooked cabbage) with oily and mild brown notes.

Table 12a: Volatile and semi-volatiles in sample Ligena 1 seeds by static headspace method

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	R.I. (Equity-5)
102	1555	carbonyl sulfide	33.43	
192	2129	acetaldehyde	45.77	
220	235	methyl mercaptan	5.05	
313	3647	ethanol	78.40	
328	123	2-propenal	2.64	
338	not determined	pentane	traces	500
339	6221	acetone	133.74	501
364	960	isopropyl alcohol	20.64	520
369	6528	dimethyl sulfide	140.34	523
393	1338	methyl acetate	28.76	541
424	3362	isobutyraldehyde	72.27	565
438	364	n-propanol	7.83	576
463	2208	diacetyl	47.47	595
470	traces	hexane	traces	600
472	756	2-butanone	16.25	602
478	372	2-methyl Furan	8.00	607
512	995	isobutyl alcohol	21.39	611
536	4108	3-methyl butanal	88.31	637
546	5752	2-methyl butanal	123.65	660
569	1981	acetic Acid	42.59	663
580	1257	pentanal	27.02	696
585	685	2-ethyl Furan	14.73	703
621	321	isoamylalcohol	6.90	739
625	627	2-methyl-1-butanol	13.48	743
633	1060	dimethyl disulfide	22.79	750
651	93034	toluene-D8(Internal Standard)	2000.00	768

Table 12b: Volatile and semi-volatiles in sample Ligena 1 seeds by purge-and-trap headspace method

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	R.I. (Equity-5)
522	93335	toluene-D8	1000.00	759
556	6208	hexanal+octane	66.51	800
621	1119	hexyl alcohol	11.99	871
643	303	2-hexanone	3.25	891
653	24957	heptanal+nonane	267.39	900
704	114	2 -heptenal(Z)	1.22	960
715	299	benzaldehyde	3.20	973

721	traces	diisoamylene	traces	980
728	270	1-butene,4-isothiocyanato	2.89	988
735	270	2-pentyl Furan	2.89	995
742	2882	octanal	30.88	1005
763	396	p-dichlorobenzene	4.24	1031
773	807	limonene	8.65	1055
774	co-eluted with 773	benzyl alcohol	traces	1056
785	1632	phenylacetaldehyde	17.49	1058
790	290	2-octenal(E)	3.11	1064
796	868	γ-terpinene	9.30	1071
825	14896	nonanal	159.60	1108
849	765	dimethyl trisulfide	8.20	1139
874		undecane-3-methyl	traces	1172
876	987	2-nonen-1-al	10.57	1175
892	traces	dodecane	traces	1200
902	4752	decanal	50.91	1210
915	2174	pyrazine,2-methyl-6-(methylthio)	23.29	1228
922	470	benzene 1,2-dimethoxy	5.04	1238
933	549	hexylcyclohexane	5.88	1253
941	394	carvone	4.22	1264
947	689	decane,2,6,7-trimethyl	7.38	1273
966	634	n-tridecane	6.79	1300
974	1059	undecanal	11.35	1312
978	764	2,2,5-trimethyl decane	8.19	1318
985	153	2,4-decadienal	1.64	1329
1028	1446	1-tetradecene+codiflex(plasticizer)	15.49	1395
1031	3076	n-tetradecane	32.96	1400
1041	2299	dodecanal	24.63	1416
1080	586	1-pentadecene	6.28	1478
1094	794	n-pentadecane	8.51	1500
1109	495	3,5-di-tert-butyl phenol	5.30	1525
1153	920	hexadecane	9.86	1600
1162	2621	codiflex(plasticizer)	28.08	1615
1181	512	tetradecane 2,6,10-trimethyl	5.49	1647
1193	341	cyclopentadecanol	3.65	1668
1209	584	1-dodecen-3-ol	6.26	1693
1212	764	n-heptadecane	8.19	1700
1288	11827	2-pentadecanone 6,10,14-trimethyl	126.72	
1375	3438	propyl hexadecanoate	36.84	

5.4.2.2: Volatiles and semi-volatile compounds in Sample Ligena 1A seeds (by static and dynamic headspace volatiles method).

A total of 68 compounds were identified in sample Ligena 1A seeds using both static and dynamic headspace volatiles technique and are listed in Tables 13a and 13b. Methyl mercaptan was identified in this sample at 27 ppb and this compound has an unpleasant rotten cabbage like odor. Dimethyl sulfide, 3-methyl butanal, 2-methyl butanal, 2-methyl propanal, acetone, acetic acid, 2-ethyl furan and acetaldehyde dominated the headspace volatiles.

In the dynamic headspace volatiles small levels of 2, 4-heptadienal, trans,2,6-nonadienal,2,4-decadienal,3, 5-octadien-2-one (fruity, green and grassy), acetophenone (fruity and sweet), 5-hepten-2-one- 6-methyl,2-pentyl furan,limonene,cis-ocimene, naphthalene,1-methyl naphthalene,γ-decalactone,carvone,2-isopropyl-3-methoxy pyrazine were observed. The major aldehydes were nonanal, heptanal and hexanal, and small levels of decanal and octanal were identified. Alcohols hexyl alcohol (green), 1-heptyn-3-ol and 1-nonanol (green, oily and floral) were also identified in this sample.

The aroma of the out-gas products from this sample can be described as fruity oily, green and mild sulfury notes.

Table 13a: Volatile and semi-volatile compounds in Ligena 1A samples by static headspace method

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	R.I. (Equity- 5)
290	3075	acetaldehyde	59.64	
307	1387	methanethiol(or methyl mercaptan)	26.90	
317	269	carbonyl sulfide	5.22	
352	4410	ethanol	85.53	
372		pentane	traces	500
375	5378	acetone	104.31	502
381	831	isopropyl alcohol	16.12	509
393	9681	dimethyl sulfide	187.76	521
407	2312	methyl acetate	44.84	534
431	10256	2-methyl propanal(same as isobutyraldehyde)	198.91	559
438	1330	n-propanol	25.80	566
462	862	diacetyl	16.72	590
469	491	2-butanone	9.52	601
505	856	isobutyl alcohol	16.60	631
529	5433	3-methyl butanal(isovaleraldehyde)	105.37	654
538	9769	2-methyl butanal	189.47	662
565	3735	acetic acid	72.44	694
575	2601	2-ethyl furan	50.45	701
607	1106	isoamylalcohol	21.45	732
611	1500	2-methyl-1-butanol	29.09	736
618	1543	dimethyl disulfide	29.93	745
636	103120	toluene D-8(Internal Standard)	2000.00	763

Table 13b: Volatiles and semi-volatiles compounds in Sample Ligena 1A seeds by Purge-and-trap method.

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	RI (Equity-5)
520	87817	toluene-d8(Internal Standard)	1000.00	769
539	283	furan,2,3-Dihydro-4-Methyl	3.22	787
544		2-hexanone	traces	792
552	14967	hexanal+octane	170.43	800
605	473	2-hexenal(E)	5.39	847
616	7764	hexyl alcohol	88.41	857
648	11636	heptanal	132.50	901
698	380	2-heptenal(E)	4.33	960
706	778	heptanol	8.86	969
709	662	benzaldehyde	7.54	973
715	1050	1-heptyn-3-ol	11.96	980

722	238	5-hepten-2-one,6-methyl	2.71	988
728	2023	2-pentyl furan	23.04	995
733	509	2,4-heptadienal	5.80	998
736	2293	octanal	26.11	1003
738		1,2,4-trimethylbenzene	traces	1005
746	554	2,4-heptadienal(E,E)	6.31	1014
757	397	p-dichlorobenzene	4.52	1029
767	3778	limonene	43.02	1039
778	2733	phenylacetaldehyde	31.12	1055
789	2624	cis-ocimene	29.88	1068
793	1202	3,5-octadien-2-one(E,E)	13.69	1073
799	411	acetophenone	4.68	1081
813	3239	undecane+2-isopropyl-3-methoxy pyrazine	36.88	1100
819	23487	nonanal	267.45	1108
860	827	trans,cis-2,6-nonadienal	9.42	1162
864	1207	2-nonenal(E)	13.74	1167
868	5964	1-nonanol	67.91	1172
883	1078	2-nonenal(E)	12.28	1192
889	2185	n-dodecane	24.88	1200
896	2153	decanal	24.52	1210
904	co-eluted	naphthalene	traces	1221
928	1304	1-dodecene	14.85	1255
938		carvone	traces	1269
959	16517	n-tridecane	188.08	1300
979	673	2,4-decadienal(E,E)	7.66	1330
984		naphthalene,1-methyl	traces	1338
1014	1400	gamma decalactone	15.94	1384
1020	4094	5-tetradecene	46.62	1392
1023		1-tetradecene+codiflex(plasticizer)	0.00	1398
1025	12774	n-tetradecane	145.46	1400
1036		4,6-dimethyl dodecane	traces	1416
1087	8212	n-pentadecane	93.51	1500
1098		dodecane-2,6,11-trimethyl	traces	1519
1103	958	phenol,2,6-bis(1,1-Dimethylethyl)	10.91	1527
1122		tetradecane,3-methyl	traces	1561
1146	2876	n-hexadecane	32.75	1600

5.4.2.3: Volatiles and semi-volatile compounds in Sample Calena 2 seeds (by static and dynamic headspace volatiles method).

A total of 65 compounds were identified in this sample and are listed in tables 14a and 14b.

The major sulfur compound identified in the static headspace volatiles was dimethyl sulfide and other compounds like acetaldehyde, acetone, ethanol, 2-methyl butanal and 3-methyl butanal dominated the static headspace volatiles. Diacetyl, dimethyl disulfide, methyl acetate, 2-methyl furan, acetic acid were also identified in this sample.

Heptanal and nonanal were found to dominate the volatiles from the purge and trap analysis and these aldehydes arise mainly from the auto-oxidation of oleic acid. 3-Thujene was found in this sample and this compound is a monoterpene with a characteristic herbal, pungent aroma found in the essential oil of some plants. Hexyl alcohol, thymol, 1-nonanol were identified in small levels and compounds like benzaldehyde, 6-methyl 5 hepten-2-one, 2, 4-decadienal and dodecanal were found in trace levels.

The aroma profile of this sample can be described as mild sulfury, fruity and oily.

Table 14a: Volatiles and semi-volatiles in sample Calena 2 seeds by static headspace method.

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	R.I. (Equity- 5)
105	3605	carbonyl sulfide	57.39	
184	2557	acetaldehyde	40.71	
202	136	dimethyl ether	2.17	
228	350	methyl mercaptan	5.57	
312	4098	ethanol	65.24	
324		2-propenal	traces	
334	6734	acetone	107.21	501
361		isopropyl alcohol	traces	519
365	7489	dimethyl sulfide	119.23	523
388	2007	methyl acetate	31.95	540
419	5338	isobutyraldehyde	84.99	563
434	319	n-propanol	5.08	573
459	1439	diacetyl	22.91	591
467	934	2-butanone	14.87	601
474	279	2-methyl Furan	4.44	602
481	654	t-butyl alcohol	10.41	608
509	527	isobutyl alcohol	8.39	634
531	7605	3-methyl butanal	121.08	655
541	8553	2-methyl butanal	136.17	664
560	1481	acetic acid+ 1-butanol	23.58	682
576	1151	pentanal+3-pentanone	18.32	697
580	1207	2-ethyl furan	19.22	701
613	910	isopentyl alcohol	14.49	736
617	528	2-methyl-1-butanol	8.41	740
625	907	dimethy disulfide	14.44	748
643	125622	toluene-d8(Internal standard)	2000.00	766

Table 14b: Volatiles and semi-volatiles in sample Calena 2 seeds by Purge-and-trap headspace method.

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	R.I. (Equity-5)
518	72570	toluene-d8	1000.00	757
550	5929	hexanal+octane	81.70	800
614	1106	hexyl alcohol	15.24	864
645	31564	heptanal	434.95	896
685	510	a-Thujene	7.03	944
703	169	trans-2-heptenal	2.33	968
707	303	benzaldehyde	4.18	972

720	180	6-methyl,5-hepten-2-one	2.48	988
726	289	2-pentyl furan	3.98	995
734	2331	octanal	32.12	1004
765	528	limonene+benzyl alcohol	7.28	1040
776	932	benezeneacetaldehyde	12.84	1052
781	348	1-heptyn-4-ol	4.80	1058
788	737	gamma-terpinene	10.16	1065
816	12330	nonanal	169.90	1098
865	785	1-nonanol	10.82	1164
890	1746	decanal	24.06	1200
899		naphthalene	0.00	1213
910	261	acetic acid, phenyl ester	3.60	1225
922	288	nonanoic acid	3.97	1243
949	622	thymol	8.57	1279
962	1535	n-tridecane	21.15	1300
970	658	2,4-decadienal(E,Z)	9.07	1312
1002	1062	2,3,4-trimethyl decane	14.63	1372
1011	1188	1-tetradecene	16.37	1387
1016	540	2,3,5,8-tetramethyl decane	7.44	1395
1019	2779	n-tetradecane	38.29	1400
1032	1372	dodecanal	18.91	1419
1078	710	1-pentadecene	9.78	1484
1089	1790	n-pentadecane	24.67	1500
1094	1141	phenol,3,5-bis(1,1-dimethylethyl)	15.72	1508
1145	4319	hexadecane	59.51	1600
1158	2850	codiflex (plasticizer)	39.27	1620
1197	5300	heptadecane	73.03	1700
1252	1715	octadecane	23.63	1800
1273	16570	2-pentadecanone,6,10,14 trimethyl	228.33	
1293	754	4-tert-octyl-o-cresol	10.39	

5.4.2.4: Volatiles and semi-volatile compounds in Sample Calena 2A seeds (by static and dynamic headspace volatiles methods)

A total of 71 volatile and semi-volatile compounds were identified in this sample using both the headspace volatiles method and are summarized in tables 15a and 15b.

The static headspace volatiles were dominated by ethanol, acetone, dimethyl sulfide, acetaldehyde, 3-methyl butanal, 2-methyl butanal and 2-methyl propanal. 2-Ethyl furan, diacetyl, methyl acetate, 2-butanone, acetic acid, isopropyl alcohol, methanethiol and dimethyl disulfide were also identified in this sample.

The dynamic headspace volatiles was dominated by heptanal, nonanal and small amounts of limonene, benzaldehyde, benzene acetaldehyde, 2-pentyl furan, 2, 4-decadienal and 2, 4-heptadienal, were also found. Compounds like 2-penty furan and 2, 4-decadienal have very low odor detection thresholds of 1 and 0.01 ppm respectively and can exert a powerful influence on the aroma profile. 2-Alkenal compounds (2-nonenal 2-decenal and 2-heptenal) and alcohols (heptanol, hexyl alcohol, 1-octanol, 1-nonanol and 2-decen-1-ol) were also identified. Aromatic compounds like 1-methyl naphthalene (6.6 ppb) and 2-methyl naphthalene (4.9 ppb) were present in very small amounts in this sample.

The odor profile of this sample can be described as oily, citrussy and mild sulfury.

Table 15a: Volatiles and semi-volatiles in sample Calena 2A identified by Static headspace method.

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	R.I. (Equity-5)
293	2632	acetaldehyde	48.93	
311	611	methanethiol	11.35	
348	5561	ethanol	103.38	
370		pentane	traces	500
372	4533	acetone	84.27	501
383	734	isopropyl alcohol	13.64	509
396	4807	dimethyl sulfide	89.36	521
411	2148	methyl acetate	39.93	536
438	4874	2-methyl propanal	90.61	562
445	626	1-propanol	11.63	568
471	782	diacetyl	14.53	592
479	551	2-butanone	10.24	601
516	757	tert-butyl alcohol	14.07	635
540	3402	3-methyl butanal	63.24	657
550	4568	2-methyl Butanal	84.92	666
577	1422	acetic acid	26.43	691
584	697	pentanal	12.95	697
589	1441	2-ethyl furan	26.78	702
624	633	isoamyl alcohol	11.76	736
627	366	2-methyl-1-butanol	6.80	739
636	572	dimethy disulfide	10.63	748
655	107579	toluene-d8(Internal standard)	2000.00	767

Table 15 b: Volatiles and semi-volatiles in sample Calena 2A identified by Purge- and -trap headspace method.

Scan #	Area Integration	Peak Assignment	Concentration PPB (w/w)	R.I. (Equity-5)
529	91528	toluene-d8	1000.00	767
548	147	furan,2,3-dihydro-4-methyl	1.61	786
562	7692	hexanal+octane	84.04	800
614	454	2-hexenal(Z)	4.96	857
625	2738	hexyl alcohol	29.91	870
656	19577	heptanal	213.89	905
706	134	2-heptenal	1.46	961
713	441	heptanol	4.82	969
717	395	benzaldehyde	4.32	973
722		1-heptyn-3-ol	traces	979
729	373	5-hepten-2-one,6-methyl	4.08	987

736	699	2-pentyl furan	7.64	990
740	326	2,4-heptadienal(E,E)	3.56	1002
743	2312	octanal	25.26	1006
753	387	2,4-heptadienal	4.23	1017
763	372	p-dichlorobenzene	4.06	1030
774	1734	limonene	18.95	1044
785	742	benezeneacetaldehyde	8.11	1060
790	450	cis-ocimene	4.92	1063
796	1229	1-octanol	13.43	1071
799	607	3,5-octadien-2-one(E,E)	6.63	1075
819	1478	undecane	16.15	1100
825	21296	nonanal	232.67	1107
870	663	2-nonenal(E)	7.24	1167
874	3880	1-nonanol	42.39	1173
889	788	isocamphane	8.61	1193
894	1211	n-dodecane	13.23	1200
902	2761	decanal	30.17	1211
909		naphthalene	traces	1221
933	838	cyclohexane,hexyl	9.16	1255
946	2635	2-decen1-ol	28.79	1273
954	515	2-decenal	5.63	1285
965	2827	n-tridecane	30.89	1300
977	4487	2,2,5-trimethyl decane+undecanal	49.02	1318
984	561	2,4-decadienal(E,E)	6.13	1329
989	607	napthalene 1-methyl+2-undecanone	6.63	1337
1002	451	naphthalene,2-methyl	4.93	1355
1018	2969	gamma-decalactone	32.44	1380
1028	2912	1-tetradecene+plasticizer(codiflex)	31.82	1397
1031	7347	tetradecane	80.27	1400
1070	1250	2-dodecanone	13.66	1464
1093	11179	n-pentadecane	122.14	1500
1104	4236	tridecane 2,5-Dimethyl	46.28	1519
1108	417	phenol,2,6-Bis(1,1-Dimethylethyl)	4.56	1525
1152	7619	n-hexadecane	83.24	1600
1161	4156	plasticizer	45.41	1616
1207	765	n-heptadecane	8.36	1700

5.4.2.5: Volatiles and semi-volatile compounds in Sample GNG-Omega meal (by static and dynamic headspace volatiles methods)

A total of 142 compounds were identified in this sample using static and dynamic headspace volatiles methods. The volatiles identified are presented in tables 16a and 16b.

The static headspace volatiles were dominated by acetaldehyde, ethanol, acetone, dimethyl sulfide, 2-methyl propanal, 3-methyl butanal, 2-methyl butanal and dimethyl disulfide. Pentanal, 2-ethyl furan, methyl acetate and acetic acid were found in moderate amounts. 1-Propanethiol, isoamyl alcohol, isobutyl alcohol and diacetyl were identified in very small quantities.

The volatiles identified by the dynamic headspace method were mainly the aldehydes, ketones, furans, acids, hydrocarbons, esters, alcohols, sulfur compounds, pyrazines, phenols, terpenes and aromatic benzene compounds. The maximum number of compounds was identified in this sample when compared to all other seed samples. The aroma and flavor compounds identified in this sample includes maillard reaction products (furfural,2-methyl propanal,3-methyl butanal and pyrazines),sulfur compounds (allyl isothiocyanate,1-butanethiol,1-propanethiol,propylisonitrile,1-butene-4-isothiocyanato and methyl propyl sulfide),naturally occurring methoxy pyrazines (2-sec-butyl-3-methoxy pyrazine) and lipid oxidation derived compounds(saturated aldehydes, 2-alkenals, 2,4-alkadienals,hydrocarbons,furan,acids and ketones). The sulfur compounds in Cruciferae family oilseeds and vegetables are formed by the action of glucosinolates on thioglycoside precursor through cooking or damaged plant tissue and result in the

formation of isothiocyanates, thiocyanates and nitriles. Allyl isothiocyanate and allyl nitrile are formed from allyl glucosinolate (Lindsay, 1996).

Limonene, 3, 5-octadiene-2-one were found in high levels. 2-pentanone, 2-heptanone, eucalyptol, phenyl ethyl alcohol, heptanol, ocimene-cis, 2, 5-dimethyl pyrazine and 2-methoxy-3-sec-butyl-pyrazine were found in small quantities. The compound 2-methoxy-3-sec-butyl pyrazine is associated with a green bell-pepper, green pea kind of note. Pentanal, heptanal, 2, 4-hexadienal and 2, 4-heptadienal was present in very small levels. 2, 4, 5-trithiahexane has a characteristic cooked cabbage odor and can be formed by the photolysis of dimethyl disulfide (Buttery and Ron, 1977). 2-Pentyl furan was identified at 730 ppb and this level was the highest when compared to all camelina seed samples.

The aroma/odor profile of this sample can be described as green (bell-pepper type), sulfury, mild fruity and citrussy. The high levels of sulfur compounds, lipid oxidation volatiles and the pyrazines (2-sec-butyl-3-methoxy pyrazine and 2-isopropyl-3-methoxy pyrazine) can affect the palatability of the omega meal when used in animal feed formulations.

Table 16a: Volatiles and semi-volatiles in sample GNG-Omega meal by static headspace method.

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	R.I. (Equity-5)
105	4036	carbonyl sulfide	192.48	
180	7199	acetaldehyde	343.32	
218	422	methanethiol	20.13	
305	7421	ethanol	353.91	
332		pentane	traces	500
334	4818	acetone	229.77	501
358	278	isopropyl Alcohol	13.26	519
365	25287	dimethyl sulfide	1205.95	524
388	2930	methyl Acetate	139.73	541
420	13025	2-methyl propanal	621.17	565
433	535	n-propanol	25.51	574
460	364	diacetyl	17.36	595
468		hexane	traces	600
470	444	2-butanone	21.17	602
475	117	2-methyl furan	5.58	606
480	208	2-butanol	9.92	610
493	98	1-propanethiol	4.67	622
508	2672	isobutyl alcohol	127.43	635
533	8954	3-methyl butanal	427.02	657
543	7671	2-methyl butanal	365.83	666
556	2285	acetic acid	108.97	678
566	754	cyclopentanol	35.96	686
578	384	pentanal	18.31	697
583	663	2-ethyl furan	31.62	701
619	2610	isoamyl alcohol	124.47	737
622	2010	2-methyl-1-butanol	95.86	740
631	5848	dimethyl disulfide	278.89	749
649	41937	toluene-d8(Internal Standard)	2000.00	766

Table 16b: Volatiles and semi-volatiles in sample GNG-Omega meal by purge-and-trap headspace method

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	R.I. (Equity-5)
298	1070	1-propanol+2-butenal	9.86	580
323	860	3-buten-2-one	7.92	596
347	762	t-butyl alcohol	7.02	615
419	4171	3-buten-2-one,3-methyl	38.43	674
421	co-eluted with419	1-hexene	traces	675

437	8745	2-penten-1-ol(Z)	80.57	689
441	27346	2-pentanone+propylisonitrile	251.93	692
447	co-eluted	1-butanethiol	traces	697
451	13357	pentanal+heptane+methyl propyl sulfide	123.06	700
456	14327	2-ethyl furan	131.99	705
466	3539	acetoin	32.60	714
513	7352	2-pentenal(E)	67.73	759
525	108544	toluene-d8(Internal standard)	1000.00	770
529	co-eluted	toluene	traces	774
534	1007	4,4-dimethyl-1-hexene	9.28	779
542	1962	furan 2,3-dihydro-4-methyl	18.08	786
547	2157	2-hexanone	19.87	790
557	44958	hexanal+octane	414.19	800
563	475	1-octene	4.38	807
567	2692	ethanol,2-(methylthio)	24.80	811
583	5047	2-methyl pyrazine	46.50	827
591	2401	furfural	22.12	836
607		cis-3-hexenol	traces	853
609	10366	2-hexenal(E)	95.50	855
612		trans-2-hexenol	traces	859
622	107878	hexyl alcohol	993.86	869
630	3716	m & p-xylene	34.23	878
639	5717	allyl iso thiocyanate	52.67	887
643	6215	2-hexanone,4-methyl	57.26	891
651	15245	2-heptanol+n-nonane+styrene	140.45	900
653	co-eluted with 651	heptanal	traces	902
654	2678	o-xylene	24.67	906
658	1235	ethanol-2-Butoxy	11.38	908
662		2,4-hexadienal(E,E)	traces	913
665	10557	2,5-dimethyl pyrazine	97.26	916
671	1719	2-ethyl pyrazine+methyl hexanoate	15.84	923
682	1279	octane,2,6-dimethyl	11.78	935
683		3-thujene	traces	936
688	2984	2-n-butyl furan	27.49	942
689	co-eluted with 688	3-carene		943
693	5702	alpha-pinene	52.53	948
695		3-hepten-1-ol	traces	951
701	Trace	nonane,2-methyl	traces	957
703	7375	trans-2-heptenal	67.94	960
708	5758	octane,2,4-dimethyl	53.05	966
711	18770	heptanol	172.93	969
714	12783	benzaldehyde+ 1,3,5-trimethyl benzene	117.77	972
721	7925	1-heptyn-3-ol	73.01	980
727		5-hepten-2-one,6-methyl	traces	988
729		1-butene,4-isothiocyanato	traces	990
731		2-heptanone,6-methyl	traces	993
734	79934	2-pentyl furan	736.42	995

738	27632	decane	254.57	1000
743	14472	octanal + 1,2,4-trimethyl benzene	133.33	1006
751	9245	2,4-heptadienal(E,E)	85.17	1016
758	14575	octane-2,5-dimethyl+ α-phellandrene	134.28	1025
762	24331	p-dichlorobenzene	224.16	1030
768	12118	p- cymene	111.64	1037
773	121317	limonene	1117.68	1043
778	9057	eucalyptol	83.44	1049
784	Trace	benzeneacetaldehyde	traces	1058
791	26294	decane,3-methyl	242.24	1065
795		γ-terpinene	traces	1070
799	76064	3,5-octadien-2-one(E,E)	700.77	1075
805	8459	2-isopropyl-3-methoxy pyrazine	77.93	1081
807		acetophenone	traces	1083
809	5835	1-undecene	53.76	1085
812		o-cymene	traces	1091
814	12971	2-nonanone	119.50	1094
819	163455	undecane	1505.89	1100
825	co-eluted with 819	nonanal	traces	1108
828	12379	5-undecene	114.05	1112
833	19151	hydrocarbon(C-12 alkane)	176.44	1118
840		3-nonen-1-ol(E)	traces	1128
841	26845	phenyl ethyl alcohol	247.32	1129
845		m-cymene	traces	1134
858	14862	2-nonenal	136.92	1151
881	5857	2-methoxy-3-sec-butyl pyrazine	53.96	1182
888	15177	cyclohexanol,1-methyl-4-(1-methylethyl)	139.82	1192
890		2-decanone	traces	1196
894	157778	n-dodecane	1453.59	1200
898	1176	6-dodecene	10.83	1206
907		naphthalene	traces	1218
924	2895	cyclopentane,1-pentyl-2-propyl	26.67	1244
933	1694	hexylcyclohexane	15.61	1256
935	2459	4-terpineol acetate	22.65	1259
942	5590	carvone	51.50	1269
955	1156	1,6-tridecadiene	10.65	1288
959	2346	1-tridecene	21.61	1293
964	42529	n-tridecane	391.81	1300
971		bornyl Acetate	traces	1312
976	10899	undecanal+decane-2,2,3-trimethyl	100.41	1319
984		2,4-decadienal	traces	1330
988	7238	1-methyl naphthalene	66.68	1336
1001	6518	2-methyl naphthalene	60.05	1357
1007	11250	undecane 3,9-dimethyl	103.64	1365
1019		γ-decalactone	traces	1385
1027		Plasticizer(codiflex)	traces	1394
1030	59842	n-tetradecane	551.32	1400

1036	coeluted	dodecanal	traces	1410
1075		2,6-di-tert butyl phenol	traces	1473
1089	2046	1-pentadecene	18.85	1494
1093	21221	n-pentadecane	195.51	1500
1100		tridecanal	traces	1512
1128		tridecanol	traces	1557
1152	7556	hexadecane	69.61	1600
1161	4071	Plasticizer	37.51	1618
1192	1329	cyclopentane,undecyl	12.24	1677
1323	11981	methyl palmitate	110.38	
1357	3348	ethyl palmitate	30.84	
1420	22036	methyl oleate	203.01	
1425	25762	methyl linolenate	237.34	

5.4.2.6: Volatiles and semi-volatile compounds in Sample Ligena 1 crushed seeds (by static and dynamic headspace volatiles methods)

A total of 78 compounds were identified in this sample using static and dynamic headspace volatiles methods and are summarizedd in Tables 17a and 17b.

The static headspace volatiles were dominated by dimethyl sulfide, dimethyl disulfide, ethanol isopropyl alcohol, isobutyl alcohol, acetic acid, acetone, and carbonyl sulfide. The compounds diacetyl, methyl acetate and 2, 4, 5-trithiahexane were also identified in this sample in small amounts.

The dynamic headspace volatiles consisted of hexanal as the major aldehyde, hydrocarbons (alkanes, alkenes),alcohols (1-nonanol,heptanol,1-octanol,hexyl alcohol and 3-hexen-1-ol), ketones(3,5-octadiene-2-one,3-octanone,cycloheptanone,4-methyl and 3-boranone). Very small levels of aldehydes (3-hexenal, decanal, octanal and benzaldehyde), and compounds like acetoin, 2-pentyl furan, 3-carene, β-pinene, limonene and β-myrcene were also identified in this sample. The methoxy pyrazines, 2-isopropyl-3-methoxy pyrazine and 2-sec-butyl-3-methoxy pyrazines were also identified in this sample (100 and 36 ppb respectively) at levels which were higher than the odor detection thresholds of these compounds. 2, 4-dithiapentane and 2, 4, 5-trithiahexane were also identified in this sample. This sample was a crushed sample and like the GNG omega meal sample, the naturally occurring pyrazines and sulfur compounds can be easily identified by aroma based on their concentration.

The aroma profile of this sample can be described as green bell-pepper, mild fruity, oily and sulfury (cabbage).

Table 17a: Volatiles and semi-volatile compounds in sample Ligena 1 crushed seeds by static headspace method.

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	R.I. (Equity-5)
107	1395	carbonyl sulfide	119.43	
186	2520	acetaldehyde	215.74	
218	645	methanethiol	55.22	
307	5526	ethanol	473.08	
330		pentane	traces	500
333	3443	acetone	294.76	501
355	2628	isopropyl alcohol	224.98	519
362	30903	dimethyl sulfide	2645.63	523
385	1677	methyl acetate	143.57	541
419	457	butanal	39.12	565
430	456	n-propanol	39.04	575
457		diacetyl	traces	597
465	353	2-butanone	30.22	602
477	203	2-methyl furan+ 2-butanol	17.38	611
489	186	carbon disulfide	15.92	632
506	1878	isobutyl alcohol	160.78	636
530	406	3-methyl butanal	34.76	658
541	1341	2-methyl butanal+acetic acid	114.80	667
617	749	isoamyl alcohol	64.12	738
620	960	2-methyl-1-butanol	82.19	741
629	7671	dimethyl disulfide	656.72	750
648	33372	toluene-d8(Internal Standard)	2857.00	769
850	426	dimethyl trisulfide	36.47	

Table 17b: Volatiles and semi-volatile compounds in sample Ligena 1 crushed seeds by Purge-and-trap method

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	R.I (Equity-5)
335	3504	hexane+2-butanone	49.94	600
419	1060	1-butanol	15.11	670
438	3042	2-penten-1-ol(Z)	43.36	687
448		2-pentanone	traces	695
454	12747	pentanal+heptane	181.67	700
465	1138	acetoin	16.22	711
513	1005	2-pentenal+pyrrole	14.32	758
524	100194	toluene-D8(Internal standard)	1428.00	769
528		toluene	traces	773
542	434	2-pentenal(Z)	6.19	786

548	963	2-hexanone	13.73	792
556	76698	hexanal+octane	1093.13	800
609		2-hexenal(E)	traces	857
611	900	cis,3-hexen-1-ol	12.83	858
621	35885	hexyl alcohol	511.45	869
629	714	m &p -xylene	10.18	878
642	1190	2-heptanone	16.96	892
650	4994	nonane	71.18	900
650	co-eluted with 650	2,4-dithiapentane	traces	901
652		heptanal+o-xylene	traces	903
686	848	2-butyl furan	12.09	941
690	592	5-methyl,1-hexanol	8.44	946
703	278	2-heptenal(E)	3.96	961
710	11088	heptanol	158.03	969
714	not determined	benzaldehyde	traces	974
720	1766	diisoamylene	25.17	980
727	3067	5-hepten-2-one,6-methyl	43.71	988
732	9136	2-penty furan+β-myrcene	130.21	994
737	1438	n-decane	20.49	1000
741	665	octanal	9.48	1005
750	846	2,4-heptadienal(E,E)	12.06	1016
754	653	1-cyclohexene-1-methanol	9.31	1020
762	367	p-dichlorobenzene	5.23	1030
772		benzyl alcohol	traces	1042
775	4247	limonene	60.53	1046
778	2045	cycloheptanone,4-Methyl -R	29.15	1050
780		3-carene	traces	1052
794	8449	1-octanol	120.42	1070
798	2498	3,5-octadiene-2-one	35.60	1074
819		undecane	traces	1100
821	7348	2-isopropyl-3-methoxy-pyrazine	104.73	1102
824	6109	nonanal	87.07	1106
827		3,5-heptadien-2-one,6-methyl	traces	1111
836	592	methyl octanoate	8.44	1123
859	630	disulfide, methyl(methylthio)methyl	8.98	1153
873	90879	nonanol	1295.24	1172
880	2546	2-methoxy-3-sec-butyl-pyrazine	36.29	1181
894	947	n-dodecane	13.50	1200
901	655	decanal	9.34	1210
909		naphthalene	traces	1221
946	774	1-decanol	11.03	1273
965	7271	n-tridecane	103.63	1300
1026	3318	n-tetradecane	47.29	1400
1093	1204	n-pentadecane	17.16	1500

5.4.2.7: Volatiles and semi-volatile compounds in Sample Ligena 1 preheated & crushed seeds (by dynamic headspace volatiles method)

A total of 61 compounds were identified in this sample using purge & trap headspace method. The headspace volatiles were dominated by aldehydes like hexanal and nonanal, alcohols (hexyl alcohol, heptanol, nonanol, isoamyl alcohol, 2-methyl-1-butanol and octanol) and sulfur compounds (dimethyl sulfide and dimethyl disulfide). Other sulfur containing compounds like 2, 4-dithiapentane, 2, 4, 5-trithiahexane, propyl isonitrile, methyl propyl sulfide were also identified in small amounts (30 ppb). The ethers 2-methyl furan, 2-butyl furan and 2-pentyl furan were identified in small to trace amounts. 7-octen-2-one, 3, 5-octadiene-2-one, 2-pentanone and 2-hexanone were also identified in this sample.

Limonene, β-myrcene, cis-ocimene and β-pinene were observed in small quantities (10-50 ppb) and these compounds are associated with fresh, green and herbal note. Both the methoxy pyrazines (2-secbutyl-3-methoxy pyrazine, 2.7 ppb and 2-isopropyl-3-methoxy pyrazine, 26 ppb) were identified in this sample at their odor detection threshold.

The aroma/odor profile of this sample can be described as sulfury, oily, mild fruity and green bell-pepper type.

Table 18: Volatiles and semi-volatiles in sample Ligena 1-Preheated & crushed seeds by purge-and-trap method

Scan #	Area Integration	Peak Assignment	Concentration PPB(w/w)	R.I. (Equity-5)
221	3333	dimethyl sulfide	38.39	526
287	3267	propanal,2-methyl	37.63	568
337	4820	hexane+2-butanone	55.52	600
344	4012	2-methyl Furan	46.21	606
349	1734	2-butanol	19.97	610
381	2640	isobutyl alcohol	30.41	637
406	934	3-methyl butanal	10.76	659
416	762	2-methyl butanal	8.78	668
420	1104	1-butanol	12.72	672
438	1202	2-penten-1-ol(Z)	13.84	686
441	690	2-pentanone	7.95	689
447	1545	propylisonitrile	17.79	694
454	6039	heptane+pentanal+methyl propyl sulfide	69.56	700
457		2-ethyl furan	traces	703
492	2878	isoamyl alcohol	33.15	737
496	3580	1-butanol,2-Methyl	41.23	741
505	951	dimethyl disulfide	10.95	750
514	520	pyrrole	5.99	759
524	123983	toluene-d8(Internal standard)	1428.00	769
528	1135	toluene	13.07	772
542	190	furan,2,3-dihydro-4-methyl	2.19	786
548	487	2-hexanone	5.61	792
556	7199	octane+hexanal	82.92	800
610	707	3-hexen-1-ol	8.14	858
620	17065	hexyl alcohol	196.55	869
628	412	m & p-xylene	4.75	877
649	3304	nonane+2,4-dithiapentane	38.05	900
651		heptanal+p-xylene	traces	903
686	537	α-thujene+2-butyl furan	6.19	943
709	13054	heptanol	150.35	969
718	1000	2-heptenal(E)	11.52	980
725	1270	5-hepten-2-one,6-methyl	14.63	987
730	4948	5-hepten-2-ol,6-methyl	56.99	993
732		2-pentyl furan+β-myrcene	traces	995
736	1100	decane	12.67	1000
740	782	octanal	9.01	1005
749		2,4-heptadienal	traces	1016
766	372	p-cymene	4.28	1037
770	3509	limonene	40.42	1042
773	300	7-octen-2-one	3.46	1045
778	813	ocimene,cis	9.36	1053

793	2779	1-octanol	32.01	1070
796	1743	3,5-octadiene-2-one(E,E)	20.08	1074
817	2312	2-isopropyl-3-methoxy pyrazine+undecane	26.63	1100
822	2846	nonanal	32.78	1108
835	631	methyl octanoate	7.27	1122
839		phenyl ethyl alcohol	0.00	1129
857	108	2,4,5-trithiahexane	1.24	1153
871	6717	nonanol	77.36	1172
879	232	2-sec-butyl-3-methoxypyrazine	2.67	1181
892	919	n-dodecane	10.58	1200
899	857	decanal	9.87	1210
907		naphthalene	traces	1227
925	150	β-cyclocitral	1.73	1247
962	11186	n-tridecane	128.84	1300
987	93	naphthalene,1-methyl	1.07	1337
1000	665	naphthalene,2-methyl	7.66	1357
1024	930	1-tetradecene	10.71	1393
1029	4407	n-tetradecane	50.76	1400

5.4.3 Identification of volatiles and semi-volatile compounds in Camelina oil.

Work on flavor and aroma compounds present in camelina oil has been very limited till date. Kirst et al., 2006 reported the volatiles in camelina oil headspace and the major compounds identified in this study were acetic acid(9.3%), trans,trans-2,4-heptadienal(3.6%), trans-2-heptenal (2.3%), α-pinene(1.6%), trans-2-pentenal(1.3%), trans-3-octen-2-one (0.8%) and trans-2-hexenal (0.2%). Compounds like butyric acid (0.1%) , isovaleric acid(0.3%), trans-2-butenal (9.8%), hexanol (0.6%), styrene (0.8%), benzaldehyde(0.4%), sabinene(0.6%), trans,trans-3,5-octadiene-2-one(3.8%), nonanal (2.2%) were also identified in their study.

Snyder and King (1994) reported the presence of C3 and C4 saturated and unsaturated aldehydes along with C2-C4 hydrocarbons in canola, corn, soybean and sunflower oil volatiles. Eight compounds were identified in major concentrations in the volatiles and they were pentane, pentanal, hexanal, 2-heptenal, 2-pentyl furan, octanal, nonanal and 2, 4-decadienal. Hexanal was found the highest in sunflower seed oil followed by soybean and corn oil and nonanal was found the highest in canola oil which had very small levels of hexanal and 2,4-decadienal. Sunflower, soybean and corn oils are rich in linoleic acid (55-75%) and canola oil is rich in oleic acid (70%).

Kirst et al., 2006 reported 54 compounds in the headspace volatiles of flaxseed oil. This oil had a very high α-linolenic acid content (45-55%) and has a fatty acid profile similar to camelina oil with the exception of eicosenoic acid. Hexanol, trans-2-butenal, acetic acid and pentanol were identified as the major compounds present in the headspace of flaxseed oils.

A total of 306 compounds were identified in camelina oil samples and are presented in Table 18. Retention indices of the sample compounds were determined based on n-alkane hydrocarbons (C-5 to C-15) present in camelina oil samples. A blank sample (with the outgas products from diatomaceous earth (Celite 545) was prepared and analyzed to ensure that the diatomaceous earth used for camelina oil sample preparation (section 4.4.1) had not picked up any aroma compounds from the storage area. Concentrations of sample volatile compounds were calculated with toluene-d8 as the internal standard and concentrations of the compounds were expressed in PPM.

Acids (10) starting from acetic to decanoic acid were identified in most of the camelina oil samples. Esters (12) were identified in the oil samples and were mostly the methyl esters of fatty acids acetic, isovaleric, oleic, plamitic, myristic and linoleic acid. Ethy acetate and octyl salicylate were also identified in some samples. Alcohols (38) were identified in camelina oil samples and the commonly observed alcohols were 1-penten-3-ol, 2-hexanol, hexyl alcohol, heptanol, 2-octen-1-ol, 2-nonen-1-ol, and trans, cis-2, 6-nonadien-1-ol. Alcohols like cis-3-hexen-1-ol (green and grassy)1-octen-3-ol (earthy and mushroom type),1-penten-3-ol (green),trans,2,6-nonadien-1-ol (green, oily and cucumber),hexyl alcohol (leafy green) and phenyl ethyl alcohol (floral and rose-like) have odor detection thresholds between 1-2500 ppb and can exert an influence on the aroma of camelina oil.

Aldehydes compounds (28) were a major group of compounds in camelina oil volatiles and are formed as a result of lipid oxidation and maillard reaction. The common aldehydes were C5-C11 saturated aldehydes, 2-alkenals, 2, 4-alkadienals, 3-methyl butanal, 2-methyl butanal, fufural and benzaldehyde. Benzaldehyde which is a

degradation product of 2, 4-decadienal originating from linoleic acid imparts a nutty almond-like flavor (Salinas et al., 1994) and was observed in all camelina oil samples.

Ketones (44) were identified in camelina oil volatiles and some of the interesting ketones were diacetyl, acetoin, 3-hexen-2-one, 3-octen-2-one and 3, 5-octadien-2-one (fatty and fruity). Ketones like diacetyl and acetoin contribute to creamy, buttery and dairy aroma.2, 6-nonadienal (green, cucumber- type), cis-4-heptenal (stale-type aroma), 3, 5-octadien-2-one and 1-octen-3-one (mushroom and fishy) are degradation products arising from linolenic acid (Belitz and Grosch, 1999) and these compounds were found in all camelina samples. Cis-geranyl acetone is an important volatile compound identified in lemon oil (Lindsay, 1996) with fresh, floral aroma and was identified in only 1 unrefined camelina sample # GNG-1.

Hydrocarbons: alkanes (30), alkenes (21) and alkynes (1) were also identified and are formed as a result of lipid oxidation. Compounds like pentane, hexanal and 2-heptenal were reported at elevated levels in aged canola oil (Shahidi and Wanasundara, 1994). Aromatic benzene compounds (19) were identified and the most common compounds were xylene, cymene, naphthalene and styrene. The aromatic hydrocarbon, p-cymene is present in significant amounts in the essential oil of certain spices (Belitz and Grosch, 1999). A total of 9 ethers compounds were identified and the furans found in all samples were 2-ethyl furan (sweet, burnt and malty), 2-butyl furan (spicy) and 2-pentyl furan (green bean and buttery). 2-Pentyl furan and isomers of 2-pentenyl furan are formed from linoleic and linolenic acids by singlet oxygen oxidation mechanism in the presence of chlorophyll (Min et al., 2003). Trace amounts of ethyl furan, tetra-hydrofuran and ethyl acetate have been identified in canola oil headspace volatiles

(Shahidi and Wanasundara, 1994). Ethyl acetate (sharp and fruity) and 2-ethyl furan were found in small to trace levels in some camelina oil samples. Phenolic (11) and quinone (4) compounds were also identified in camelina oil samples. Anti-oxidants BHA and BHT were identified in some samples. Most of the phenols contribute to a smoky and woody aroma. The quinone 2, 6-di-tert butyl benzoquinone (DBQ) was identified in most of the samples.

A total of 17 terpene and sesquiterpene compounds have been identified in camelina oil samples. The sesquiterpenes and oxygenated sesquiterpenes were mostly found only in unrefined camelina oils. Mono and sesquiterpenes are found in fruits, vegetables and spices and are usually associated with pleasant (floral, herbaceous, citrus, camphor-like and woody) aromas (Belitz and Grosch, 1999). Terpenes like β-myrcene, cis-ocimene are associated with fresh, leafy green and floral aromas and have been identified only in unrefined camelina samples and not detected in refined and deodorized samples. Limonene was identified in all camelina samples.

Nitrogen containing compounds (12) were identified and the naturally occurring 2-sec-butyl-3-methoxy pyrazine was found in unrefined and deodorized camelina samples. 2-Sec-butyl-3-methoxy pyrazine has been identified as one of the typical aroma substance in carrot and also identified in parsley (Belitz and Grosch, 1999) and red beets (Lindsay, 1996). Compounds like 2-acetylpyrrole (nutty and musty), 2, 3, 5-trimethyl pyrazine (roasted and nutty) and 2, 3, 4, 5-tetramethyl pyrazine (nutty and roasted, brown type) were identified in some samples.

Six (6) sulfur containing compounds were identified and the most common sulfur compounds were dimethyl sulfoxide and dimethyl sulfone.Dimethyl disulfide and

dimethyl trisulfide were identified only in unrefined camelina oils and dimethyl sulfone was identified in all camelina oil samples. Most of the sulfur compounds are aroma-active and contribute a typical sulfury (onion, cabbage and cauliflower) aroma. 1-Butene-4-isothiocyanato and 2, 4, 5-trithiahexane was also identified in some samples. Several glucosinolates found in Cruciferae family give rise to characteristic flavors and produce isothiocyanates, nitriles and thiocyanates (Lindsay, 1996). Compounds like 2-nonenitrile and 2-undecenitrile were identified in some unrefined camelina oils.

Compounds like ethylene glycol, diethylene gylcol butyl ether and 1-methoxy 2-butanol was also identified in most of the oil samples and these compounds are typical solvents used in the chemical and oil industry.

Volatile and semi-volatiles in unrefined camelina oils: The presence of aldehydes, acids, ketones, hydrocarbons, terpenes, sulfur containing compounds (dimethyl disulfide, dimethyl sulfone) and nitrogen containing compounds (pyrazines) were observed (Tables 22,23,24 and 25). Dimethyl sufone is an oxidation product of dimethyl sulfide. Aromatic benzene compounds like ethylbenzene, o, m & p-xylene, p-ethyl toluene, 1, 3, 5-trimethyl benzene, p-cymene and naphthalene and were observed in some samples. Vanillin (Sweet, creamy and balsamic aroma) was also identified in 1 sample GNG-1 at 0.06ppm. 5-Pentyl resorcinol was identified in samples GNG-18 and 19 and 4-tert-amyl phenol was found in 2 samples GNG-1 and GNG-18. The phenol compounds are generally formed from phenolic acids and lignin due to thermal degradation or by the action of micro-organisms in the food where they are detected (Belitz and Grosch, 1999). They are mostly responsible for smoky, woody aroma in food

products like coffee, oilseeds and some nuts. Anti-oxidants BHA and BHT were identified in sample GNG -1 and 18.

Compounds like β-zingiberene, caryophyllene, isocaryophyllene, β-bergamotene, β-farnesene, β-himachalene, patchoulene, germacrene b and curcumene were found mostly in small amounts (0.2-0.5 ppm) in unrefined oil samples GNG-1 and 19. Curcumene and β-zingiberene were also reported in the leaves of wild tomato and function as insect-repellant (Antonious & Kochhar, 2003). Curcumene has also been identified in ginger oil during storage due to the odixation of zingiberene (Belitz and Grosch, 1999). Beta-Himachalene has been identified in the essential oil of dried Nigerian ginger and has a spicy and fresh aroma (Onyenekwe & Hashimoto, 1999). β-Zingiberene and caryophyllene were reported in considerable amounts in rhizome oil of Zingiber nimmoni (Sabulal et al., 2006) and these compounds have been reported to possess anti-microbial activity. They also act as insecticides, repellants and insect-feeding deterrents (Antonious & Kochhar., 2003). β-Farnesene is a constituent of various essential oils and is emitted by plants as a natural insect repellant. The presence of such sesquiterpene compounds probably helps camelina plants in self-defense mechanism against insects and weeds. 2-Butenal, hexanal, 2-heptenal, 2, 4-heptadienal and 2, 4-decadienal were found as the major compounds in the volatiles of unrefined camelina oil samples.

Volatile and semi-volatiles in deodorized camelina oils: Aldehydes,acids, alcohols, hydrocarbons,ketones,conjugated dienes and trienes,aromatic benzene compounds, terpenes and pyrazine compounds were identified (Tables 26 and 27). 2-Pentyl furan, benzalehyde, limonene and β-farnesene were found in all deodorized samples in small

levels (0.2-5.0ppm). Dimethyl sulfone was identified in both the deodorized samples (0.15 ppm). The compound 2-sec-butyl-3-methoxy pyrazine was identified only in 1 deodorized sample, # GNG-13(deodorized 1 time) at 0.08 ppm and was not present in the other deodorized oil sample GNG-12(deodorized 3 times). 5-Pentyl resorcinol and 4-tert-amyl phenol were identified in both the samples. Coumarin (or 2H-1-benzopyran-2-one), which has a sweet, mild hay-like aroma was identified in both the deodorized samples in small levels (0.3-0.5ppm). The anti-oxidant BHA was identified in sample GNG-13. Synthetic anti-oxidants like BHA, BHT and a mixture of BHA/ BHT/ MGC (monoglyceride citrate) have been used in the canola oil industry, but use of natural anti-oxidative compounds like flavanoids, phenolic acids, lignans, terpenes, phospholipids and polyfunctional acids should help in replacing or reducing the levels of the synthetic anti-oxidants (Shahidi and Wanasundara,1994). 1-Penten-3-ol, 1-octen-3-ol,2,4-heptadienal, 2,4-decadienal,2-butenal,2-pentenal,2-heptenal, 2-octenal,2-decenal,2-nonenal, nonanal, hexanal,3,5-octadien-2-one and trans,cis-2,6-nonadien-1-ol were found to dominate the headspace volatiles of deodorized camelina oils.

Volatile and semi-volatiles in refined camelina oils: The number of compounds identified in refined oils was much lower than unrefined and deodorized camelina oil samples (Tables 20, 28 and 29). This is expected since the last step in refining vegetable oils is the deodorizing process, which helps in removing the volatile compounds formed due to lipid oxidation and maillard reactions. 2-pentyl furan was found at 0.9 ppm in sample GNG-4 and this is low when compared to all other camelina oil samples. Ethyl acetate, furfural and 2-ethy furan were identified in both the refined oil samples in small amounts. Also dimethyl sulfone was found in sample GNG-22 at 0.25 ppm. The

compound 2-methoxy-3-methyl pyrazine was not detected in refined oil volatiles, suggesting that it could have removed or reduced to very low levels during the refining process. 2, 6-di-tert butyl benzoquinone was found in both the samples in trace levels (0.05 ppm). 1-Penten-3-ol,2-pentenal,hexanal,2-heptenal,2-octenal,2,4-heptadienal, nonanal,3,5-octadien-2-one,2-pentanone, and 2,4-decadienal were found to dominate the headspace volatiles of refined oil samples .

Table 19: Volatile and semi-volatile compounds identified in camelina oil samples

Total number of compounds identified:306	
Acids(12 identified)	
acetic acid	butanoic acid
propionic acid	isovaleric acid
pentanoic acid	octanoic acid
hexanoic acid	nonanoic acid
heptanoic acid	decanoic acid
2-methyl hexanoic acid	propanoic acid,2,2-dimethyl
Alcohols(40 identified)	
isoamyl alcohol	2-methyl-1-butanol
4-pentyn-2-ol	2-hexen-1ol
1-octanol	2-hexanol
1-penten-3-ol	hexyl alcohol
4-heptyn-3-ol,1-heptyn-3-ol	7-octen-4-ol
2-nonen-1-ol	1-decyn-4-ol
E,Z-2,6-nonadien-1-ol	2-octen-1-ol
2-butanol	1-nonanol
1-hexadecanol	heptanol
α-terpineol	1,6-dihydrocarveol
bicyclo(3.1.1) heptan-3-ol,2,6,6-trimethyl	3-nonyn-2-ol
β-terpineol	cis-carveol
farnesol	trans,carveol
n-propanol	phenyl ethyl alcohol
1-nonen-4-ol	borneol
cis-p-menth-1-en-3-ol	4-hexen-1-ol
cis-verbenol	1-ccten-3-ol
1-pentanol	Furfuryl alcohol
1-butanol	2-penten-1-ol
2-hepten-1-ol	5-hexen-1-ol
Esters(12 identified)	
methyl ester of pentanoic acid	methyl palmitate
2-hexen-1-ol,acetate(E)	methyl acetate
methyl oleate	ethyl acetate
octyl salicylate	ethyl plamitate
methyl myristate	methyl linoleate
methyl,trans,cis-farnesate	methyl isovalerate
Phenolic compounds(11 identified)	
6-propyl-m-cresol	5-pentyl resorcinol
6-methyl-2,4-di-tert-butyl phenol	3-ethyl phenol
2-tert butylated hydroxy anisole(or 2-BHA)	1-naphthalenol,4-methoxy
4-hexanoyl resorcinol	2,6-di-tert-butyl-4-methyl phenol(or BHT)
phenol,4-(1,1-dimethylpropyl)	phenol,2,6-Bis(1,1-dimethylethyl)

4n-propyl resorcinol	
Quinone compounds(4 identified)	
2,5-di-tert butyl benzoquinone	2,3-dimethyl hydroquinone
2(1H)-quinoxaline	2,6-di-tert-butylbenzoquinone(or DBQ)
Carbonyl compounds-aldehydes(33 identified)	
2-methyl propanal	2-propenal
2-butenal	2-pentenal
3-methyl butanal	2-methyl butanal
2-hexenal,3-hexenal	4-heptenal
hexanal	heptanal
octanal	nonanal
benzene acetaldehyde,alpha-methyl	2-octenal
2,4-nonadienal	2,6-nonadienal
2-undecenal	cyclopentanecarboxaldehyde
p-ethylbenzaldehyde	2,4-decadienal
furfural	2,5-dimethylbenzaldehyde
2,4-hexadienal	2,4-heptadienal
4-decenal	2-furyl-3-methylbutanal
2-decenal	5-ethyl-2-furaldehyde
β-cyclocitral	2-heptenal
benzaldehyde	trans-2-dodecen-1-al
3-furaldehyde	
Carbonyl compounds-ketones(49 identified)	
3-buten-2-one	3-penten-2-one
2-heptanone	2-butanone
2-octanone	2-decanone
cyclohept-4-enone	2-octanone,3-octanone
cycloheptanone,4-methyl	3,5-octadien-2-one
4-isopropenylcyclohexanone	4-isopropenylcyclohexanone
3-decen-5-one	butyrophenone,2',4',5'-trihydroxy
α-ionone	cis-geranyl acetone
4,5,7,7a-tetrahydro-4,4,7a-trimethyl-2(6H)benzofuranone	2(4H)-benzofuranone,5,6,7,7a-tetrahydro-4,4,7a-trimethyl(or Dihydroactinidiolide)
3,5-heptadien-2-one,6-methyl	3-hexen-2-one,5-hexen-2-one
2-nonanone	2'-hydroxy-4',5'-dimethyl acetophenone
2,3-pentanedione	2,5-furandione,3,4-dimethyl
diacetyl	acetoin
1-penten-3-one	2-hexanone
3,4-dimethoxyaxetophenone	ethanone,1-(2,2-dimethylcyclopentyl)
2H-1-benzopyran-2-one(same as coumarin)	2-undecanone
trans-3-octen-2-one	2-pentanone
2(3H)-benzofuranone,3a,4,5,6-tetrahydro-3a,6,6-trimethyl	ethanone,1,1-(6-hydroxy-2,5-benzofurandiyl)bis-(or euparone)
megastigmatrienone	2-Octen-4-one
1H-pyrrole,2,5-dione,3-Ethyl-4-methyl	camphor

aristol-9-en-8-one	carvone
p-mentha-1,8-dien-3-one(+)	2-methylacetophenone
5,6-dihydro-2-pyranone	3-isopropyl-4a,5-dimethyloctahydro-1(2H)-naphthalenone
2-pentadecanone-6,10,14-trimethyl	2(3H)furanone,dihydro-4,4-dimethyl
γ-decalactone	
Hydrocarbon-alkanes(30 identified)	
hexane,2,3,5-trimethyl	heptane,3-ethyl
octane,2,2-dimethyl	nonane,2,6-dimethyl
decane,3,7-dimethyl	6,8-dioxabicyclo 3.2.1 octane
pentane	hexane
heptane	octane
nonane	decane
undecane	dodecane
tridecane	tetradecane
pentadecane	decane,3-methyl
cyclopentane,2-isopropyl-1,3-dimethyl	cyclohexane,pentyl
bicyclo 3.3.1 nonane	octane,2,6-dimethyl
heptane,2,4,6-trimethyl	nonane,2,5-dimethyl
nonane,3-methyl	nonane 3,7-dimethyl
nonane 2,6-dimethyl	octane,2,5-dimethyl
decane,2,4,6-trimethyl	cyclopentane
Hydrocarbon-alkenes(21 identified)	
1,3-pentadiene	2-pentene
1-pentene,3-ethyl-2-methyl	4-octene,1-octene
1,4-heptadiene,3-methyl	2,4-octadiene
1,3,6-octatriene(E,E)	cis-4-decene
1,4-hexadiene-3-ethyl	1,5-cyclodecadiene
p-mentha-1,3,8-triene	1,3,5-undecatriene
2-undecene,7-methyl	5-tetradecene,7-tetradecene
5-undecene	1-dodecene
diisoamylene	4-tertbutylcyclohexene
1-octene,7-methyl	1-nonene
1-tridecene	
Hydrocarbon -aromatics(21 identified)	
1,3,5-trimethyl benzene	p-ethyl toluene
benzene-2,4-diethyl,1-methyl	1H-cyclopropa A naphthalene, 1A ,2, 3, 3A, 4, 5, 6,7B -octahydro 1,1,3A tetramethyl
2,4-diethoxy-1,5-formylbenzene	ethyl benzene
benzene-1,2-diethyl	naphthalene,decahydro
m-xylene,2-ethyl	1,2,3,5-tetramethyl benzene
p-ethyl cymene	p-ethyl styrene
naphthalene	styrene
1,2,3-trimethyl benzene	p-propyl toluene
o-xylene	p-cymene
naphthalene decahydro,2-methyl	naphthalene,1-Methyl

benzene,(1-methylethyl)	
Ethers(12 identified)	
2-propyl furan	ethyl-2-benzofuran
2-heptyl furan	2-ethyl furan
2-hexyl furan	2-pentyl furan
2-butyl furan	2-methyl furan
furan,2,2'-(1,2-ethenediyl)bis-(E)	furan,2,3-dihydro-4-methyl
benzoyl ethyl ether	2-(2-propenylfuran)
Sulfur compounds(6 identified)	
dimethyl sulfone	dimethyl sulfoxide
dimethyl disulfide	dimethyl trisulfide
1-butene-4-isothiocyanato	disulfide,methyl(methylthio)methyl
Terpene compounds(18 identified)	
limonene	sabinene
β-pinene	2-carene
cis-ocimene	β-Myrcene
β-zingiberene	caryophyllene
β-bergamotene	β-farnesene
β-himachalene	curcumene
β-bisabolene	germacrene B
isocaryophyllene	patchoulene
caryophyllene oxide	α -copaene
Nitrogen containing compounds(12 identified)	
2,3,5-trimethyl pyrazine	2-sec-butyl-3-methoxy pyrazine
2-nonenenitrile,2-undecenenitrile	n-methyl piperidine
1H-pyrrole,2,5-dihydro	2-acetylpyrrole
isobutyrlnitrile	2,6-diethyl pyrazine
2-methoxy pyrazine	2,5-dimethyl pyrazine
2,3,5,6-tetramethyl pyrazine	6-methyl-2-pyrazinyl methanol
Miscellaneous compounds (17 identified)	
2-methyl-1-nonene-3-yne	2H-1-benzopyran-2-one
cycloheptene,5-ethylidene-1-methyl	2-propyl tetrahydropyran
diethylene glycol	diethyl phthalate
2-(5-methylfuran-2-yl)-propionaldehyde	cis-linalool oxide
diethylene glycol butyl ether	propanol,1-(2-methoxypropoxy)(solvent)
ethylene glycol	6-heptene-2,4-diol
1H-pyrrole,2,5-dione,3-Ethyl-4-Methyl	limonene oxide
1-methoxy-2-butanol	2-butoxy-1-ethanol
1,3-benzodioxole,5-propyl(or dihydrosafrole)	
Unknown compounds(8)	
unknown ,mw 135,bp 84	unknown,mw 150,bp 41

unknown,mw 162,bp 161	unknown,mw 208,bp 138
unknown,mw 166,bp 123	unknown,mw 152,bp 69
unknown ,mw 152,bp 135	unknown,mw 160,bp 159

Table 20: Total number of volatile compounds identified in camelina oil samples.

S.No	Sample description	Type	Method	Total no of compounds
1	Camelina GNG 1	Unrefined	P&T-TD	194
2	Camelina GNG19	Unrefined	P&T-TD	171
3	Camelina GNG17	Unrefined	P&T-TD	156
4	Camelina GNG 18	Unrefined	P&T-TD	165
5	Camelina GNG-12	Deodorized*	P&T-TD	154
6	Camelina GNG-13	Deodorized*	P&T-TD	171
7	Camelina GNG-4	RBD**	P&T-TD	123
8	Camelina GNG-22	RBD**	P&T-TD	131

* Deodorized samples (GNG-12-deodorized 3 times, GNG-13-deodorized 1 time)

**Refined samples (chemically refined -degummed, neutralized, bleached, winterized and deodorized)

Table 20 lists the number of volatile compounds identified in each camelina oil sample. It can be observed that the least number of volatiles was identified in refined camelina oil samples (GNG-4 and 22). The deodorization process helps in removing the undesirable volatile compounds arising due to the oxidation of oil (Nawar, 1996). Deodorized camelina oil sample GNG-12 had lesser number of volatile compounds when compared to sample GNG-13 since sample GNG-12 had been deodorized three times. Samples GNG 12 and 13 were deodorized versions of unrefined camelina oil GNG-1, and the deodorization process alone has been helpful in removing 20 and 12% of the total number of volatile compounds from the original sample # GNG-1. Deodorized sample GNG-12 did not contain the 2-sec-butyl-3-methoxy pyrazine but this pyrazine was identified in the other deodorized sample GNG-13 at 0.08ppm. Unrefined camelina oil samples GNG-1, 17, 18 and 19 had more number of volatiles compounds than refined and deodorized samples.

5. 4.3.1 Lipid oxidation compounds in camelina oil volatiles.

Table 21: Key oxidation products identified in camelina oil samples

	Unrefined	Unrefined	Unrefined	Unrefined	Deodorized	Deodorized	Refined	Refined
Compound	**GNG -17**	**GNG-18**	**GNG-1**	**GNG-19**	**GNG-12**	**GNG-13**	**GNG-4**	**GNG-22**
propanal	n.d	0.02	0.13	n.d	n.d	0.055	0.25	0.5
2-butenal	4.15	2.5	1.35	5.39	6.34	4.8	2.54	6.46
1-penten-3-ol	7.24	2.68	0.87	1.84	10.18	5.83	9.59	6.7
pentanal	1.6	traces	0.55	0.49	1.28	1.45	3.09	6.9
2-pentenal	4.8	5.76	0.95	0.99	7.9	8.17	5.755	10.39
2-ethylfuran	2.03	1.02	0.27	0.22	0.88	0.52	0.39	2.81
hexanal	5.17	2.38	11.54	7.51	7.02	7.25	10.62	7.19
2-hexenal	2.15	2.39	0.95	1.25	3.13	3.96	1.33	3.4
2,4-hexadienal(E,E)	1.2	1.45	0.44	1.09	1.85	1.51	0.37	0.64
2-heptenal	15.6	10.62	4.01	7.9	18.54	18.7	5.97	9.46
heptanal	3.94	2.07	0.73	0.7	2.55	1.8	0.4	1.037
2-pentylfuran	3.13	3.37	3.05	3.08	3.96	3.7	0.9	3.52
octanal	11.6	2	0.56	0.74	3.96	4.29	4.73	2.09
2,4-heptadienal(E,E)	14.48	14.31	15.6	13.68	27.6	33.07	26.1	11.05
1-octen-3-ol	0.86	n.d	0.672	n.d	traces	n.d	n.d	n.d
3,5-octadien-2-one	5.5	6.975	2.85	2.58	8.005	11.7	4.8	4.16
2-octenal	20.43	18.44	6.49	6.66	31.72	35.37	7.87	11.46
nonanal	3.47	2.08	4.6	5.45	13.65	8.44	9.44	6.82
(E,Z)-2,6-nonadien-1-ol	4.63	5.97	2.29	2.6	4.08	7.27	1.15	0.91
2-nonenal	9.07	9.2	2.25	1.62	10.2	10.82	2.4	1.68
2,6-nonadienal	2.2	0.5	0.64	0.84	2.74	3.35	1.04	0.94
2,4-nonadienal(E,E)	3.07	3.62	1.49	1.71	3.63	2.08	0.7	0.5
decanal	0.32	0.28	0.36	0.73	0.76	0.67	0.16	0.2
2-decenal	8.57	6.78	4.07	6.95	11.5	20.91	3.8	2.85
2,4-decadienal	27.27	29.24	11.25	10.45	26.3	18.9	18.02	11.07
2-undecenal	1.8	0.71	0.15	0.6	3.95	3.56	1.06	0.79

n.d-not detected

Table 21 shows some of the key oxidation products found in the headspace volatiles of camelina oil. The major oxidation products identified were 1-penten-3-ol, 2-pentenal, hexanal, 2-heptenal, 2, 4-heptadienal, 2-octenal, 3, 5-octadien-2-one, nonanal, 2-nonenal, 2, 4-decadienal, and 2-decenal. Compounds like 2-pentyl furan,2,4-heptadienal,3,5-octadien-2-one,2-octenal,nonanal,2-nonenal,2-decenal,2,4-decadienal and 2-undecenal were identified at very high levels in deodorized samples. Most of these

compounds like 2,4-decadienal(0.07 ppb),2-decenal(0.4 ppb),2-heptenal(13 ppb) , hexanal(5 ppb),cis-3-hexenal(0.25 ppb),2,6-nonadienal(0.01 ppb),2,4-nonadienal(0.1 ppb),nonanal(1 ppb),2-nonenal(0.1 ppb),1-octen-3-ol(1 ppb) and 2-pentyl furan(6 ppb) have very low odor detection thresholds (indicated in parenthesis) and can impart off-flavor to camelina oil. Conjugated diene and triene compounds(1,3-pentadiene,2,4-octadiene,1,3-octadiene,1,3,5-octatriene and 1,3,5-undecatriene) were also identified in most of the camelina oil samples. Lipid oxidation studies use conjugated dienes as primary lipid oxidation product indicators and propanal and hexanal concentrations as secondary lipid oxidation products indicators. The volatile oxidation products of vegetable oils such as propanal, pentanal, pentanol, hexanal, octanal, 1-octen-3-ol can be detected in the human mouth at their odor detection threshold and can impart off-flavor to the vegetable oil (Van Ruth et al., 2000).

The oxidative stability of vegetable oils depends on their fatty acid composition and the levels of anti-oxidants in the oil (mainly tocopherols and some other non-saponifiable constituents). Among the non-saponifiables, the chlorophyll pigments are note-worthy. Chlorophyll pigments act as anti-oxidants in the dark but are singlet O_2 sensitizers and pro-oxidants in the presence of light. Chlorophyll pigments belong to the type II sensitizer group and hence once activated in the presence of light, react with the ground state triplet oxygen. The ground state triplet oxygen is transformed into singlet-state oxygen, which reacts with unsaturated fatty acids by cyclo-addition mechanism (Belitz and Grosch, 1999). Sizova et al., (2003) had reported the presence of carotenoids and chlorophyll in camelina oil using UV spectroscopy. The presence of chlorophylls in camelina oil could also be responsible for the high levels of 2-pentyl furan in camelina oil

volatiles since it was also reported that 2-pentyl furan concentrations increased in soybean oil when light exposure time and added chlorophyll level increased (Min et al., 2003).

Camelina oil observed in our study followed a lipid oxidation pattern which is similar to other vegetable oils rich in oleic, linoleic and linolenic acids. The fatty acids oleic,linoleic,α-linolenic are quite high in camelina oil(Table 3) and as seen from Table 4, the samples used for volatile compounds analysis were abundant in α-linolenic acid content (15.6-27.31%). The rate of oxidation of oleic: linoleic: linoleic acids are of the order of 1:40-50:100 and the rate of peroxide development is 1:12:25 in vegetable oils (Frankel, 1997). The hydro peroxides formed are thermally unstable and degrade to form various secondary oxidation products which can affect the sensory and nutritive quality of the edible oil. Eicosenoic acid probably undergoes an oxidation pattern similar to oleic acid since the degradation products of oleic acid, namely 2-octenal, 2-decenal, octanal, nonanal and heptanal were found in high amounts. Most of the oxidation products observed in this study on camelina oil volatiles were identified at concentrations higher than their odor detection threshold and the aroma profile of unrefined camelina oils on organoleptic evaluation at room temperature was green, nutty, earthy and oily. The aroma of the refined oils was very mild and oily suggesting that some of the compounds contributing to green and earthy aroma were present at very low concentrations or probably removed during the refining process.

Some of the volatile oxidation products identified in camelina oil were also observed in the volatiles of camelina seeds and meal in small concentrations.

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5.4.4: Identification of volatile compounds in individual camelina oil samples.

This section will detail the volatile and semi-volatile compounds identified in individual camelina oil samples.

5.4.4.1 Identification of volatiles and semi-volatile compounds in Sample GNG 17 unrefined Camelina Oil

The volatiles and semi-volatiles identified (156) in sample GNG 17 are summarized in Table 22. The major volatiles identified in this sample were the aldehydes (saturated and unsaturated), ketones, furans, hydrocarbons (alkanes, alkenes, alkynes), alcohols and acids. Small levels of sulfur compounds (dimethyl disulfide and dimethyl sulfone (1.3 ppm) and 2-sec-butyl-3-methoxy pyrazine (0.05 ppm) were also identified in this sample. The sulfur and pyrazine compounds have very low detection threshold but can easily exert an influence in the aroma of the oil which might be objectionable in edible oil.

The major aldehydes identified were 2,4-decadienal(27 ppm), 2-heptenal (16ppm), 2,4-heptadienal(14.5 ppm), 2-octenal(20.4ppm), octanal(12 ppm), 2-nonenal (9.07 ppm), 2-decenal(8.6 ppm),2-butenal(4.15 ppm) and hexanal(5.20 ppm). Dimethyl sulfone (1.32ppm), 2-pentyl furan (3.13ppm), benzaldehyde-α-methyl (3 ppm) and 1-pentanol (1.30ppm) were found in small amounts. Compounds like ethyl acetate, acetic acid,acetoin,3-octen-2-one,sabinene,benzaldehyde,phenyl ethyl alcohol,l-carvone,1-nonanol,2-butyl furan,3-hexene-2-one,1-methyl naphthalene,cis-verbenol,dimethyl disulfide,n-methyl piperidine and 7-octen-4-ol were found in very small concentrations.

Appearance and aroma: Color of the oil was golden yellow and had oily, nutty, mild earthy and green aroma.

Table 22: Volatile and semi-volatile compounds in unrefined camelina oil GNG-17 by Purge-and-trap headspace method

Scan #	Area Integration	Peak Assignment	Concentration PPM(w/w)	R.I (Equity-5)
190	213	1,3-pentadiene(E)	0.02	531
243	1141	4-pentyn-2-ol	0.09	564
258	593	2-butenal(E)	0.05	573
287	225	2-methyl propanal	0.02	591
288	co-eluted with 287	2-pentene	traces	592
293	3547	2-butanone	0.28	595
301	1788	hexane	0.14	600
334	1370	ethyl acetate	0.11	621
345	2276	1,3-butadiene,2,3-dimethyl	0.18	627
358	4902	acetic acid	0.38	636
385	51968	2-butenal(E)	4.10	652
420	3690	1-butanol	0.29	674
442	91912	1-penten-3-ol	7.25	687
445	co-eluted with 442	2-pentanone	traces	689
459	19914	pentanal	1.57	697
463	18216	heptane	1.44	700
467	25809	2-ethyl furan	2.03	702
486	629	acetoin	0.05	713
530	779	3-penten-2-one(E)	0.06	740
539	9029	2-pentenal(E)	0.71	745
541	co-eluted with 539	isoamyl alcohol	traces	745
549	2846	2-butenal,3-methyl(E)	0.22	751
558	48845	2-pentenal isomer	3.85	756
570		dimethyldisulfide	traces	763
572	50748	toluene-d8(Internal standard)	4.00	764
579	4914	toluene	0.39	768
586	16219	1-pentanol	1.28	772
590	6095	2-penten-1-ol	0.48	775
600	1614	2-hexanol	0.13	781
608	811	furan,2,3-dihydro-4-methyl	0.06	786
618	4801	2-hexenal+4-octene(E)	0.38	791
622		butanoic acid	traces	793
633	170924	octane	13.47	800
640	65698	hexanal	5.18	804
645	13122	4-octene(Z)	1.03	807
652	1663	3-hexenal(Z)	0.13	811
657	8590	2,4-octadiene	0.67	814
659	co-eluted with 657	3-octene(E)	traces	815
669	3026	1,4-heptadiene,3-methyl	0.24	822
676	1676	1,3-octadiene	0.13	826
681	1067	2-hexen-1-ol(Z)	0.08	828

693	3771	2-hexene,2,5-dimethyl+n-methyl piperidine	0.29	835
702	7053	3-hexen-2-one	0.56	841
710	21562	2-hexenal(E)	1.69	846
742	4036	hexane,2,3,5-trimethyl	0.32	865
750	3026	hexyl alcohol	0.24	869
755	11576	heptane,3-ethyl	0.92	872
758	co-eluted	m-xylene		873
767	1838	1,3,6-octatriene(E,E)	0.15	879
784	6020	2-heptanone	0.47	889
786	co-eluted with 784	pentanoic acid		890
790	2530	1-ethyl-3-methyl cyclohexane(C,T)	0.19	893
795	4196	o-xylene	0.33	896
802	49932	nonane	3.94	900
803	co-eluted with 802	heptanal		901
818	15253	2,4-hexadienal(E,E)	1.20	910
831	1032	heptane 2,4,6-trimethy	0.08	918
837	co-eluted	4-heptyn-3-ol		921
838	16764	dimethyl sulfone	1.32	922
843	co-eluted with 838	branched c-10 hydrocarbon		925
848	2779	2(3H)furanone,dihydro-4,4-dimethyl +benzene,(1-methylethyl)	0.22	928
858	23539	octane 2,6-dimethyl	1.85	934
863	3433	n-butylcyclopentane	0.27	937
871	19713	octane,2,5-dimethyl	1.55	942
876	4573	2-butyl furan	0.36	946
882	1225	2-hepten-1-ol(E)	0.09	949
898	197911	2-heptenal(E)	15.60	959
904	co-eluted with 908	octane,2,4-dimethyl	traces	963
908	56000	nonane,3-methyl	4.41	965
910	co-eluted with 908	benzaldehyde	traces	966
919	59841	hydrocarbon(C-10)	4.71	972
922	co-eluted with 919	1,3,5-trimethylbenzene	traces	974
929	9863	heptanol+cis-4-decene	0.77	978
935	25163	1-isopropyl-3-methylcyclohexane	1.98	983
935		7-octen-4-ol	traces	983
945	23107	1-methyl-2-propylcyclohexane	1.82	988
954	39737	2-pentyl Furan	3.13	993
963	88208	2,4-heptadienal(E,E)	6.95	999
966	co-eluted with 963	hexanoic acid + 1,2,3-trimethyl benzene	Traces	1001
974	147079	octanal	11.59	1006
990	183669	2,4-heptadienal(E,E)	14.48	1017
997		nonane,2,5-dimethyl	traces	1021
1002		nonane,2,6-dimethyl	traces	1024
1009	10244	sabinene+2-octanone	0.81	1029
1014	31848	p-cymene	2.51	1032
1016	co-eluted with 1014	trans 3-octen-2-one	traces	1035
1019	6497	decane,3-methyl	0.51	1036

1024	co-eluted	2-octen-1-ol(E)		1038
1027	56322	cyclohexane,pentyl	4.44	1041
1034	10868	1-octen-3-ol	0.86	1046
1044		cycloheptanone,4-methyl		1052
1046	8242	branched C 11 hydrocarbon	0.65	1054
1053	39368	benzeneacetaldehyde,α-methyl	3.10	1058
1058	70882	t-pentylcyclohexane	5.58	1062
1062	co-eluted with 1058	2-methylacetophenone	traces	1064
1064	259288	2-octenal(E)	20.43	1066
1069		naphthalene, decahydro	Traces	1069
1078	69790	3,5-octadien-2-one(E,E)	5.50	1075
1090	24239	4-isopropenylcyclohexanone	1.91	1083
1098	12746	p-cymene	1.00	1088
1102	5855	2-nonanone	0.46	1094
1127	44053	nonanal	3.47	1108
1133	23912	2-nonen-1-ol(E)	1.88	1112
1142	8801	m-xylene,2-ethyl	0.69	1118
1144		ethyl-2-benzofuran	traces	1119
1151	not determined	phenyl ethyl alcohol	traces	1125
1156	13020	decane,3,7-dimethyl	1.02	1128
1161		p-mentha-1,3,8-triene	traces	1131
1186	58712	trans,cis-2,6-nonadien-1-ol	4.62	1148
1191	115148	2-nonenal(E)	9.07	1152
1201	27909	2,6-nonadienal(E,Z)	2.19	1158
1221	11929	1-nonanol	0.94	1172
1225	co-eluted with 1221	p-ethyl benzaldehyde	traces	1175
1229	715	2-sec-butyl-3-methoxy pyrazine	0.06	1178
1248		octanoic acid	traces	1191
1250	33352	diethylene glycol butyl ether	2.62	1192
1265		naphthalene	traces	1202
1270	4119	decanal	0.32	1206
1276	1283	cyclononanone	0.10	1209
1288	39013	2,4-nonadienal(E,E)	3.07	1215
1313	1048	3-decyne+unknown compound,mw 139,bp139	0.08	1230
1325	31480	2-decenal +3-decen-5-one	2.48	1236
1340	1229	l-carvone	0.09	1247
1352	100947	2-decenal(Z)	7.96	1251
1361		nonanoic acid	traces	1256
1366	30156	terpenoid	2.38	1259
1394	8950	terpenoid	0.71	1274
1397	345976	2,4-decadienal(E,E)	27.27	1276
1416	1703	1-methyl naphthalene	0.13	1287
1439	3656	tridecane	0.29	1300
1447	7855	cis-4-decenal	0.62	1309
1456	3594	3-undecene,9-methyl	0.28	1320
1464	32460	decane,2,4,6-trimethyl	2.56	1329

1469		2-heptyl furan	traces	1335
1472	4752	unknown compound,mw 208,bp 138	0.37	1338
1484	22605	trans,2-undecenal	1.78	1352
1501	2488	unknown compound,mw 160,bp 159	0.19	1372
1512	2000	7-tetradecene	0.16	1385
1571	486	2'-hydroxy-4',5'-dimethylacetophenone	0.04	1440
1590	6891	phenol,3,5-di-tert-butyl	0.54	1458
1613	20320	trans-2-dodecenal	1.60	1478
1625	2085	2,6-di-tert-butylbenzoquinone	0.16	1489
1641	2356	pentadecane	0.18	1500
1646	not determined	α-ionone	traces	
1714	1399	4,5,7,7a-tetrahydro-4,4,7a-trimethyl-2(6H) benzofuranone	0.11	
1721	1140	butyrophenone,2',4',5'-trihydroxy-	0.09	
1745	4112	benzoyl ethyl ether	0.32	
1751	5871	cis-verbenol	0.46	
1813	687	euparone	0.05	
1954	1556	2-pentadecanone-6,10,14-trimethyl	0.12	
1991	950	methyl palmitate	0.07	

5.4.4.2 Identification of volatiles and semi-volatile compounds in Sample GNG 19 unrefined Camelina Oil

A total of 171 compounds were identified in this sample and are presented in table 23. 2-heptenal (18ppm), 2, 4-heptadienal (13.6 ppm) and 2, 4-decadienal (10.5 ppm) were found to dominate the headspace volatiles. The major fatty acids were α-linolenic (16%), linoleic (9.8%),oleic (9.7%),eicosenoic (12.3%) and erucic (5.09%)(Table 4). 2-Butenal(5.4ppm),3,5-octadien-2-one(2.58 ppm),nonanol (2.42 ppm),β-farnesene (0.72 ppm) and β-begamotene(3.73ppm) were found in small amounts. 2-Ethyl furan,1-penten-3-ol,acetic acid, iso amyl alcohol,2-pentenal,hexyl alcohol,2-heptanone,2,4-hexadienal, benzaldehyde,heptanol,beta-pinene and limonene were found in levels between 0.15 to 2 ppm.

Phenyl ethyl alcohol,1-propanethiol,2-methyl butanal,acetoin,butanoic acid,3-hexen-2-one,3-hexen-1-ol,1-butene-4-isothiocyanato,β-myrcene,2-methoxy pyrazine were found in trace quantities(0.05 ppm). 2-Secbutyl-3-methoxy pyrazine, 3-carene, carvone, 1, 6-dihydrocarveol, 3-decen-5-one, γ-decalactone, geranyl acetone and dihydroactinidiolide were also identified in this sample (0.10-0.5 ppm). Sesquiterpene compounds like caryoplhyllene, isocaryophyllene, β-zingiberene, germacrene b and β-bisabolene and curcumene were also identified (0.1-0.7 ppm).

Appearance and aroma: Color of the oil was bright golden yellow and had an oily, nutty, sweet and green vegetable(bell-pepper) type aroma.

Table 23: Volatile and semi-volatile compounds in unrefined camelina oil GNG-19 by Purge-and-trap-headspace method.

Scan #	Area Integration	Peak Assignment	Concentration PPM(w/w)	R.I(Equity-5)
101	541	propanal,2-methyl	0.05	
117	236	2-propenal,2-methyl	0.02	
150	633	2-butanone	0.05	
158	842	hexane	0.07	600
189	597	1-propanethiol	0.05	620
210	6730	acetic Acid	0.56	634
238	64442	2-butenal(E)	5.38	652
243	3092	3-methyl butanal	0.25	656
257	760	2-methyl butanal	0.06	665
264	1021	1-butanol	0.08	670
272	6372	3-penten-2-one	0.53	673
293	21967	1-penten-3-ol+2-pentanone	1.83	687
309	5868	pentanal	0.49	697
313	5860	heptane	0.49	700
316	2567	2-ethylfuran	0.21	702
327	465	2-pentanol	0.04	709
334	662	acetoin	0.06	714
346	1278	propanoic acid	0.11	721
377	4705	isoamyl alcohol	0.39	739
382	not determined	2-methyl-1-butanol	traces	742
387	3741	2-pentenal	0.31	745
394	1521	2(5H)-furanone	0.12	749
404	6522	2-pentenal(E)	0.54	755
420	42696	toluene-D8(Internal Standard)	3.57	764
432	11961	cyclopentanecarboxaldehyde	1.00	771
436	736	1-pentanol	0.06	774
454	1638	2-penten-1-ol	0.13	785
467	8284	2-hexanol	0.69	792
476	not determined	butanoic acid	traces	798
481	89861	octane+hexanal	7.51	800
493	11000	4-octene(E)	0.92	808
506	13518	2,4-octadiene+3-octene(E)	1.13	815
514	647	propanoic acid,2,2-dimethyl	0.05	820
525	7833	1,3-octadiene	0.65	826
537	199	3-furaldehyde	0.02	833
541	430	3-hexen-2-one	0.04	835
551	721	dimethyl sulfoxide	0.06	841
559	8064	3-pentenal,4-methyl	0.67	846
571	7648	2-hexenal(E)	0.64	853
574	not determined	5-hexen-2-one	traces	855
578	232	3-hexen-1-ol	0.02	857

586	1271	2-methyl hexanoic acid	0.11	862
595		2-hexen-1ol (Z)	traces	867
600	19335	hexyl alcohol	1.62	870
604	co-eluted with 600	p-xylene	traces	872
616	5792	1,3,6-octatriene	0.48	879
633	4261	2-heptanone	0.36	889
637		pentanoic acid	traces	891
645	566	cyclopentane,1-ethyl-2-propyl-	0.05	896
652	8057	heptanal+nonane	0.67	900
662	1033	2-butoxy-1-ethanol	0.09	906
668	13010	2,4-hexadienal(E,E)	1.09	909
672	traces	2,5-dimethylpyrazine	traces	912
681	696	dimethyl sulfone	0.06	917
685	traces	3-ethyl phenol	traces	919
689	545	methyl isovalerate	0.06	926
707	2030	1,3,6-heptatriene,5-methyl-(E),	0.17	932
713	2209	ethane,1-Methoxy-2-(methoxy methoxy)-	0.18	936
716	traces	sabinene	traces	938
720	1926	2-butyl furan	0.16	940
735	654	3-hepten-2-ol+ dimethyl trisulfide	0.06	949
747	96116	2-heptenal(E)	8.04	956
753	1508	benzaldehyde	0.13	963
768	6014	heptanol	0.50	969
776	2635	1-hepten-3-ol	0.22	974
779	5290	1-heptyn-3-ol	0.44	975
785	16441	branched C-10 hydrocarbon	1.37	979
790	11294	β -pinene	0.94	982
791	co-eluted with 790	1-butene,4-isothiocyanato	traces	982
792	co-eluted with 790	5-hepten-2-one,6-methyl	traces	983
799	traces	2-Octanone	traces	987
801	co-eluted with 803	β-myrcene	traces	988
803	36947	2-pentylfuran	3.09	989
807	traces	hexanoic acid	traces	992
814	99913	2,4-heptadienal(E,E)	8.35	995
818	traces	2-methoxy pyrazine	traces	997
821	8880	octanal	0.74	999
826	co-eluted with 821	2,3,5-trimethyl pyrazine	traces	1003
838	63765	2,4-heptadienal	5.33	1011
857	1731	benzyl alcohol	0.15	1022
864	traces	p-cymene	traces	1028
870	55328	d-limonene	4.63	1033
878	5520	3-carene	0.46	1038
883	traces	cycloheptanone,4-methyl	traces	1041
891	5900	cis-ocimene	0.49	1047
906	79686	2-octenal(E)	6.66	1056
910	co-eluted with 906	2-hexen-1-ol,acetate(E)	traces	1059
918	8437	2-octen-1-ol(E)	traces	1063

925	30856	3,5-octadien-2-one(E,E)	2.58	1069
932	2000	3-oxatricyclo(5.2.0(2,4))nonan-8-one	0.17	1074
938	3000	heptanoic acid	0.25	1077
941	2012	2-ethyl,3,5-dimethyl pyrazine	0.17	1080
952	1981	1-ethyl-2-cylohexanol	0.17	1087
976	65167	nonanal	5.45	1103
980	6852	3,5-heptadien-2-one,6-methyl	0.57	1106
999	13544	phenyl ethyl alcohol	1.13	1119
1005	7827	trans,p-2,5-menthadien-1-ol	0.65	1123
1012	20582	limonene oxide+ hydrocarbon	1.72	1126
1034	31117	trans,cis-2,6-nonadien-1-ol	2.60	1143
1039	38249	2-nonenal(Z)	3.19	1147
1049	10073	2,6-nonadienal(E,Z)	0.84	1154
1054		camphor	traces	1157
1069	co-eluted with 1071	phenol,2-methoxy	traces	1167
1071	28903	nonanol	2.42	1169
1075		octanoic acid	traces	1172
1077	1006	2-sec-butyl-3-methoxy pyrazine	0.08	1173
1094	8345	1-nonen-4-ol	0.69	1185
1099	7703	ethanol,2-(2-butoxyethoxy)-,	0.64	1188
1102		2-decanone	traces	1190
1107	6170	2,4-nonadienal(E,E)	0.52	1194
1113	traces	naphthalene	traces	1198
1117		1,6-dihydrocarveol	traces	1201
1119	8783	decanal	0.73	1202
1124	3901	cis-carveol	0.33	1207
1137	20402	2,4-nonadienal(E,E)	1.71	1216
1151	9961	trans-carveol	0.83	1227
1161	1373	1H-pyrrole,2,5-dione,3-ethyl-4-methyl	0.12	1235
1174	10543	3-decen-5-one	0.88	1245
1180	2622	cis-4-decenal	0.22	1250
1187	8327	l-carvone	0.69	1255
1202	83093	2-decenal(Z)	6.95	1245
1206		nonanoic acid	traces	1269
1210	1727	1-dodecene	0.14	1273
1215	9411	cycloheptene,5-ethylidene-1-methyl	0.78	1277
1222	4433	unknown compound,mw 150,bp 41	0.37	1282
1224	co-eluted with1222	5-dodecene	traces	1284
1227	6073	p-mentha-1,8-dien-3-one-(+)-	0.51	1287
1229	traces	terpenoid	traces	1288
1241	17549	terpenoid	1.47	1297
1245	55278	2,4-decadienal(E,E)+tridecane	4.62	1300
1278	70042	2,4-decadienal isomer	5.86	1324
1287	881	cis-linalool oxide	0.074	1333
1295	3019	2-pentene-1-Butoxy	0.25	1340
1312	12699	branched C-13 hydrocarbon	1.06	1353
1320	2471	unknown compound,mw 208,bp 138	0.21	1360

1333	7128	trans-2-undecenal	0.59	1370
1338	not determined	γ-decalactone	traces	1374
1344		tridecane,3-methyl	traces	1379
1353	6064	phenol,3,5-dimethyl acetate	0.51	1386
1361		7-tetradecene	traces	1392
1371	1347	tetradecane+unknown mw 162,bp 161	0.11	1400
1374	993	β-zingiberene	0.08	1403
1381		2-undecanone	traces	1412
1385	traces	1H-cyclopropa A naphthalene, 1A,2,3,3A, 4,5,6,7B -octahydro1,1,3A tetramethyl (or β-maaliene)	traces	1412
1388	664	4-hydroxy-2-methoxy benzaldehyde	0.06	1414
1431	573	caryophyllene	0.05	1450
1436	44562	β-bergamotene	3.73	1454
1441	2038	cis-geranyl acetone	0.17	1458
1446	8530	β-farnesene	0.71	1462
1450		2H-1-benzopyran-2-one(or coumarin)	traces	1466
1461	4012	2-dodecenal	0.33	1475
1469	1681	3',5'-dimethoxyacetophenone	0.14	1482
1471		2(3H)-benzofuranone-3a,4,5,6-tetrahydro-3a,6,6-trimethyl	traces	1484
1473	851	2,6-di-tert-butylbenzoquinone	0.07	1485
1482	3939	3-pentadecene	0.33	1493
1488	12251	curcumene	1.02	1497
1491		pentadecane	traces	1500
1498	9188	isocaryophyllene	0.77	
1503	2672	germacrene B	0.22	
1518	1914	β-bisabolene	0.16	
1526	3009	1-naphthalenol,4-methoxy	0.25	
1533	574	cis-farnesol	0.05	
1538	1238	5-pentylresorcinol	0.10	
1548	204	furan,2,2'-(1,2-ethylidenediyl)bis-,(E)	0.02	
1566	3286	dihydroactinidiolide	0.27	
1596	1909	dihydrosafrole	0.16	
1604	2101	2,4-undecadienal	0.18	

5.4.4.3 Identification of volatiles and semi-volatile compounds in Sample GNG 1 unrefined Camelina Oil

A total of 194 compounds were identified in this sample and are presented in Table 24. 2, 4-decadienal (12 ppm), 2, 4-heptadienal (16 ppm), hexanal, 2-heptenal (4 ppm), 2-octenal (6.5ppm), nonanal (4.6ppm), 2-decenal (4 ppm) and β-bergamotene were found to dominate the headspace volatiles of this sample. Saturated aldehydes (like pentanal, heptanal, octanal and decanal) were present in smaller amounts. The major fatty acids in this oil were α-linolenic (23%), oleic and linoleic, each at 15% and eicosenoic acid (13.4%) (Table 4). 2-Pentyl furan, phenyl ethyl alcohol, acetic acid,β-pinene,cis-ocimene,3,5-octadien-2-one,1-nonanol,,β-farnesene and curcumene were found in moderate levels(1-3 ppm).

2-Undecenal, γ-decalactone**,** 2-sec-butyl-3-methoxy pyrazine, 2-acetyl pyrrole, 2, 5-dimethyl pyrazine, 2-butyl furan, 2-tert butylated hydroxyl anisole (or BHA) were found in very small levels (0.06-0.5 ppm). Dimethyl sulfone, dimethyl trisulfide, 2-methyl furan, acetone, propanoic acid, isovaleric acid, furfuryl alcohol, furfural, styrene, sabinene, isobutyronitrile, benzaldehyde, megastigmatrienone, and patchoulene were also identified in this sample in trace amounts (0.05-0.15ppm). Vanillin, cis-farnesol, 2-nonene nitrile and methyl myristate were also identified at very low concentrations (0.05 ppm).

Appearance and aroma: Color of the oil was golden yellow and had an oily, nutty, and mild green bell-pepper kind of aroma.

Table 24: Volatiles and semi-volatile compounds in Sample GNG 1 unrefined camelina Oil by Purge-and-trap headspace method

Scan #	Area Integration	Peak Assignment	Concentration PPM(w/w)	R.I.(Equity-5)
124	302	2-propenal	0.03	
141	2649	pentane + propionaldehyde	0.22	500
148	355	acetone	0.03	503
162	967	2-pentene(E)	0.08	513
193	902	1,3-pentadiene(E)	0.07	532
246	385	propanal,2-methyl	0.03	564
262	280	2-propenal,2-methyl	0.02	573
270	295	n-propanol	0.02	574
291	448	methyl vinyl ketone	0.04	590
297	2226	2-butanone	0.18	594
306	1931	hexane	0.16	600
315	482	2-methylfuran	0.04	606
322	184	methyl cyclopentene	0.02	608
351	15988	acetic acid	1.29	628
363	5383	methyl acetate	0.44	632
389	16676	2-butenal(E)	1.35	651
396	2889	3-methyl butanal	0.23	656
411	1301	2-methyl butanal	0.11	665
422	337	1-butanol	0.03	672
426	2521	3-buten-2-one,3-methyl	0.21	674
445	10654	1-penten-3-ol	0.87	686
447	co-eluted with 445	2-pentanone	traces	687
462	6710	pentanal	0.55	697
467	3324	heptane	0.27	700
471	3355	2 ethyl Furan	0.27	702
488	1026	acetoin	0.08	712
512	4324	propionic acid	0.35	726
533	3478	isoamyl alcohol	0.28	739
539	2256	2,methyl-1-butanol	0.18	742
541	3000	dimethyl disulfide	0.24	744
542	6573	2-pentenal(E)	0.53	744
546		2-pentanol	traces	745
559	5195	2-pentenal isomer	0.42	754
576	45937	toluene-D8(Internal Standard)	3.73	764
582	1116	toluene	0.09	768
587	5255	1-pentanol+2-hexenal(E)	0.42	771
591	1871	2-penten-1-ol(Z)	0.15	773
602	1244	2-hexanol	0.10	784
618	408	2-hexanone	0.03	789
621	4701	1-octene	0.38	790
624	traces	butanoic acid	traces	791

636	142072	octane	11.54	800
639	co-eluted with 636	hexanal		801
648	4394	4-octene(E)	0.36	807
660	13810	2,4-octadiene	1.12	814
670	traces	propanoic acid,2,2-dimethyl	traces	820
680	2043	1,3-octadiene	0.17	825
691	638	furfural + n -methyl piperidine	0.05	832
705	634	3-hexen-2-one	0.05	840
709	982	dimethyl sulfoxide	0.08	843
713	11550	2-hexenal(E)	0.94	845
719	1329	isovaleric acid	0.11	846
731	2156	furfuryl alcohol	0.17	856
740	1649	isobutyronitrile	0.13	861
754	18840	hexyl alcohol	1.53	869
770	2671	1,3,6-octatriene(E,E)	0.22	879
780	1151	1-methoxy-2-butanol	0.09	885
784	4026	pentanoic acid	0.33	887
788	1063	2-heptanone	0.09	888
794	470	4-hexen-1-ol +styrene	0.04	893
799	636	o-xylene	0.05	896
802	950	4-heptenal(Z)	0.07	898
806	8940	nonane + heptanal	0.73	900
816	763	2-butoxy-1-ethanol	0.06	907
821	5416	2,4-hexadienal(E,E)	0.44	910
825	4207	2,5-dimethyl pyrazine	0.34	912
833	1077	dimethyl sulfone	0.08	917
839	663	2,5-dimethyl-2,5-dihydrofuran	0.05	921
841	co-eluted with 839	3-ethyl phenol	traces	922
843	733	methyl isovalerate	0.06	923
861	993	1,3,6-heptatriene,5-methyl(E)	0.08	935
866	1249	ethane,1-Methoxy-2-(methoxy methoxy)(solvent)	0.10	938
870	502	sabinene	0.04	940
874	6079	2-butyl furan	0.49	943
879	3540	1-heptyn-3-ol	0.29	946
883	1005	1,4-hexadiene,3-ethyl	0.08	948
900	49372	2-heptenal(E)	4.01	959
911	1874	benzaldehyde	0.15	966
921	5647	heptanol	0.46	972
931	3542	dimethyl trisulfide	0.29	979
936	not determined	hexanoic acid	traces	982
939	12645	hydrocarbon(C-10)+1-butene,4-isothiocyanato	1.03	984
943	13940	β pinene	1.13	987
953		2-octanone	traces	989
955	37521	β-myrcene+2-pentylfuran	3.05	994
966	119382	2,4-heptadienal(E,E)	9.69	1002
974	6912	octanal	0.56	1006

975	co-eluted with 974	2,3,5-trimethyl pyrazine	traces	1008
992	75062	2,4-heptadienal(E,E)	6.09	1018
1003	1632	2-carene	0.13	1025
1010	2561	benzyl alcohol	0.21	1030
1017	1941	p-cymene	0.158	1035
1023	55733	d-limonene	4.53	1039
1033	8279	1-octen-3-ol	0.67	1045
1041		cycloheptanone,4-methyl-R	traces	1051
1045	16028	cis-ocimene	1.30	1053
1060	79917	2-octenal(E)	6.49	1063
1063	1339	2-hexen-1-ol acetate	0.11	1065
1070	5059	2-acetyl pyrrole	0.41	1070
1074		2-octen-1-ol(E)+n-octanol	traces	1072
1079	35066	3,5-octadien-2-one(E,E)	2.85	1076
1085		heptanoic acid	traces	1079
1088	13606	hexenoic acid isomer +2,3,5,6-tetramethyl pyrazine	1.11	1082
1095	10179	2,6-diethyl pyrazine	0.83	1086
1107	951	2-nonanone	0.077	1094
1129	56624	nonanal	4.60	1108
1133	1714	3,5-heptadien-2-one,6-methyl-(E)-	0.14	1111
1152	17400	phenyl ethyl alcohol	1.41	1115
1163	8382	trans-P-2,8-menthadien-1-ol	0.68	1130
1172	1358	1,3,8-p-menthatriene	0.11	1136
1177	1202	disulfide, methyl(methylthio)methyl	0.09	1139
1187	28256	trans,cis2,6-nonadien-1-ol	2.29	1146
1192	17770	2-nonenal	1.44	1149
1195		2-nonen-1-ol	traces	1152
1203	7814	trans,cis2,6-nonadien-1-al	0.63	1156
1206		camphor	traces	1158
1211	10708	2-nonenal(E)	0.87	1161
1224	30605	1-nonanol	2.48	1170
1227		p-ethylbenzaldehyde	traces	1172
1230	4526	octanoic acid	0.37	1173
1232	co-eluted with 1230	2-sec-butyl-3-methoxypyrazine	traces	1174
1235	2206	1-nonen-4-ol	0.18	1177
1238	831	borneol	0.07	1179
1248	2408	unknown compound, mw 152,bp 69	0.19	1186
1253	8394	2-decanone	0.68	1189
1267	1589	alpha-terpineol+ naphthalene	0.13	1197
1272	4423	1,6-dihydrocarveol	0.36	1201
1274	2461	decanal	0.20	1204
1275	3950	1,5-cyclodecadiene(E,Z)+ unknown compound mw 152,bp 135	0.32	1203
1281	8064	cis-carveol	0.66	1208
1290	18303	2,4-nonadienal(E,E)	1.48	1215
1300	714	cis-β-terpineol	0.06	1223

1304	8663	trans-carveol	0.70	1226
1314	2249	1H-pyrrole,2,5-dione,3-ethyl-4-methyl	0.18	1233
1325	3067	3-decen-5-one	0.25	1242
1333	2717	cis-4-decenal	0.22	1245
1341	9837	1-carvone	0.79	1254
1354	50169	2-decenal(E)	4.07	1264
1356	co-eluted with 1354	nonanoic acid	traces	1266
1361	4542	1-undecene	0.37	1269
1368	15753	terpenoid	1.28	1275
1375	6996	2-nonenenitrile	0.57	1280
1382	7258	cycloheptene,5-ethylidene-1-methyl	0.59	1285
1395	44226	terpenoid	3.59	1296
1397	56250	2,4-decadienal(E,E)	4.57	1297
1402	600	**tridecane**	0.05	1300
1430	92916	2,4-decadienal isomer	7.54	1324
1433	co-eluted with 1430	terpenoid	traces	1327
1466	8664	3-decen-2-one	0.70	1352
1474	2507	unknown compound,mw 208,bp 138	0.20	1359
1486	3929	trans-2-undecenal	0.32	1369
1492	2344	γ-decalactone	0.19	1373
1498		unknown compound,mw 160,bp 159	traces	1378
1507	9672	1-tridecene	0.78	1386
1509		2-undecenenitrile	traces	1387
1515	1869	7-tetradecene	0.15	1392
1517		2-undecenal	traces	1394
1525	1595	tetradecane+alpha-copaene	0.13	1400
1528	1572	β-zingiberene	0.13	1403
1535	283	2-undecanone	0.03	1409
1539	1419	β-maaliene(1H-cyclopropa A naphthalene,1aa,2,3,3a,4,5,6,7b-a'-octahydro-1,1,3aa',7-tetramethyl	0.12	1412
1542	688	vanillin	0.06	1415
1584	856	caryophyllene	0.07	1451
1590	53103	β-bergamotene	4.31	1455
1595	1095	cis-geranylacetone	0.09	1460
1600	29316	(Z)-beta-farnesene	2.38	1464
1604	coeluted	2H-1-benzopyran-2-one	traces	1467
1623	4225	3,4-dimethoxy acetophenone	0.34	1484
1639	7017	β-himachalene	0.57	1497
1642	18190	pentadecane	1.48	1500
1644	co-eluted with 1642	curcumene	traces	
1652	14721	isocaryophyllene	1.19	
1657	5897	sesquiterpene	0.48	
1665	732	patchoulene	0.06	
1675	4531	BHT(anti-oxidant)	0.37	

1680	3998	1-naphthalenol,4-methoxy	0.33	
1692	1535	sesquiterpene	0.13	
1697	538	farnesol	0.04	
1702	357	furan,2,2'-(1,2-Ethenediyl)bis-,(E)	0.03	
1719	1901	dihydroactinidiolide	0.15	
1732	757	BHA(anti-oxidant)	0.06	
1750	667	phenol,4-(1,1-dimethylpropyl)	0.05	
1756	995	megastigmatrienone	0.08	
1776	337	caryophyllene oxide	0.03	
1824	500	oxygenated sesquiterpene	0.04	
1865	395	methyl myristate	0.03	
1941	2635	octyl salicylate	0.21	
1954	2211	2-pentadecanone-6,10,14-trimethyl	0.18	
1977	1411	1-hexadecanol	0.11	
2002	1895	methyl palmitate	0.15	
2101	2294	methyl oleate	0.18	
2105	1248	methyl linoleate	0.10	

5.4.4.4 Identification of volatiles and semi-volatile compounds in Sample GNG 18 unrefined Camelina Oil

A total of 165 compounds were identified in this sample and are listed in Table 25. 2-Heptenal (10 ppm), 2,4-heptadienal (17.7ppm), 3,5-octadien-2-one (7 ppm), 2-octenal (18.4 ppm), nonanal (8 ppm), 2-nonenal (12.5ppm), 2,6-nonadien-1-ol (6 ppm), 2-decenal (6.9 ppm) and 2,4-decadienal (29.2 ppm) were found to dominate the headspace volatiles of this sample. 2-Pentylfuran, 1-octen-3-ol, dimethyl sulfone, 2, 4-nonadienal, heptanal, octanal, benzaldehyde, trans-3-octen-2-one, 2-methyl acetophenone, 1-penten-3-ol and acetic acid were identified in small levels (3 to 5 ppm).

Compounds like 2-butanone, 2-ethyl furan, 2-pentenal, 3-hexenal, hexanal, 1-pentanol and 1-nonanol were identified in very small levels (1 to 3 ppm). Trace amounts of methyl vinyl ketone, 2-propenal, propanal, 3-penten-2-one, dimethyl sulfoxide, phenyl ethyl alcohol, 2-sec-butyl-3-methoxy pyrazine, β-ionone, isocaryophyllene, dihyrdo-actinidiolide, 6-propyl-m-cresol, 5-pentyl resorcinol and octyl salicylate were also identified.

Appearance and aroma: Color of the oil was golden yellow and had an oily, nutty, and mild green bell-pepper kind of aroma.

Table 25: Volatiles and semi-volatiles in unrefined camelina oil sample GNG-18 by purge-and-trap headspace method

Scan #	Area Integration	Peak Assignment	Concentration PPM(w/w)	R.I.(Equity-5)
131	94	2-propenal	0.02	
137	317	cyclopentane	0.04	
147	732	pentane	0.08	500
158	164	propanal	0.02	507
166	177	2-pentene(E)	0.02	512
196	502	1,3-pentadiene(E)	0.06	531
286	597	methyl vinyl ketone	0.07	587
298	2153	2-butanone	0.24	595
306	905	hexane	0.10	600
367	12112	acetic acid	1.36	638
389	22211	2-putenal(E)	2.49	651
397	1300	2-propenal,2-methyl	0.15	656
425	3522	3-puten-2-one,3-methyl	0.39	673
446	23922	1-penten-3-ol	2.68	686
449	386	2-pentanone	0.04	689
463	10503	pentanal	1.18	697
468	4662	heptane	0.52	700
471	9097	2-ethylfuran	1.02	702
482	1151	acetoin	0.13	708
507	2460	propionic acid	0.27	723
534	694	3-penten-2-one(E)	0.08	739
543	25792	2-pentenal(E)	2.89	744
548	503	2-butenal,3-methyl	0.06	747
576	34314	toluene-d8(Internal Standard)	3.85	764
582	5707	toluene	0.64	767
587	11237	1-pentanol	1.26	770
606	2378	2-penten-1-ol(Z)	0.27	782
621	4071	1-octene	0.46	790
633	1245	butanoic acid	0.14	798
637	100657	octane+hexanal	11.29	800
649	8209	4-octene(E)	0.92	807
651	1334	2-hexanol	0.15	808
656	892	3-hexenal(Z)	0.10	811
661	12029	2,4-octadiene	1.35	814
673	2402	3-octene+1,3-heptadiene,3-methyl	0.27	822
680	2324	1,3-octadiene	0.26	826
685	764	5-hexen-1-ol	0.09	829
699	2463	dimethyl sulfoxide+ hydrocarbon	0.28	836
705	2948	3-hexen-2-one	0.33	841
714	29224	2-hexenal(E)	3.28	846
733	traces	pyrazine,2,6-dimethyl	traces	857

745	3738	isobutyronitrile	0.42	865
753	3338	hexyl alcohol	0.37	869
758	9992	p-xylene	1.12	872
771	2533	1,3,6-octatriene,(E,E)	0.28	880
778	389	1-methoxy-2-butanol	0.04	885
788	5230	2-heptanone	0.59	890
790	917	pentanoic acid	0.10	894
799	4358	o-xylene	0.49	897
805	10039	nonane	1.13	900
808	26818	heptanal	3.00	902
821	12934	2,4-hexadienal(E,E)	1.45	910
836	21976	dimethyl sulfone	2.47	920
846	454	octane 2,6-dimethyl	traces	926
851	2168	benzene,(1-methylethyl)-	0.24	929
861	10873	2-heptenal(E)	1.22	935
863	coeluted with 863	1,3,5-heptatriene,5-methyl	traces	937
866	3392	ethane,1-methoxy-2-(methoxy methoxy)	0.38	938
874	15763	2-butyl furan	1.77	943
882	6967	1-heptyn-3-ol	0.78	947
899	87151	2-heptenal(E)	9.78	959
907	16814	octane,2,4-dimethyl	1.89	964
911	38300	benzaldehyde+ hydrocarbon(C-10)	4.29	966
913		m-ethyl toluene	traces	967
922	26133	1-heptanol+hydrocarbon	2.93	973
925		1,3,5-trimethyl benzene	traces	975
932	3751	branched C-10 hydrocarbon	0.42	984
943	12515	p-Ethyl toluene	1.40	987
946	15852	p-menthane, cis	1.78	989
948	co eluted	hexanoic acid	traces	989
956	30047	2-pentyl Furan	3.37	994
960		2-octen-4-one	traces	997
965	56586	2,4-heptadienal	6.35	1000
969		2-hepten-1-ol	traces	1003
975	26741	octanal	3.00	1007
992	100806	2,4-heptadienal(E,E)	11.31	1017
995		o-propyl toluene	traces	1020
1005	33852	nonane,2,6-dimethyl	3.798	1026
1011	7758	benzyl alcohol + p-cymene	0.87	1030
1016	25242	benzene,1,2-diethyl+trans-3-octen-2-one	2.83	1034
1022	4332	d-Limonene	0.49	1038
1027		cyclohexane,butyl	traces	1041
1030	34160	1-octen-3-ol+branched C-11 hydrocarbon	3.83	1043
1037	8188	cycloheptanone,4-methyl	0.92	1048
1053	6430	benzene,1,3-diethyl	0.72	1058
1056	31408	benzeneacetaldehyde,alpha,methyl	3.52	1060
1061	164227	2-octenal(E)	18.43	1063
1064		2-hexen-1-ol acetate	traces	1065

1066	55998	hydrocarbon(C-11)+2-methylacetophenone	6.28	1067
1072	11347	2-octen-1-ol+naphthalene,decahydro	1.27	1071
1080	62169	3,5-octadien-2-one(E,E)+hydrocarbon	6.98	1076
1082	co-eluted with 1080	p-propyl-toluene	traces	1078
1085	not determined	heptanoic acid	traces	1080
1090	10201	4-isopropenylcyclohexanone	1.15	1083
1097	16603	3-ethyl,o-xylene	1.86	1087
1106	6300	5-undecene	0.71	1093
1108		1,3,8-p-menthatriene	traces	1094
1124	6389	m-menth-3(8)-ene	0.72	1105
1130	68681	nonanal	7.71	1109
1136	9000	benzene-2,4-diethyl,1-methyl	1.01	1114
1138	co eluted	3,5-heptadien-2-one,6-methyl,(E)-	traces	1122
1155	2555	phenyl ethyl alcohol	0.29	1127
1157	11867	naphthalene decahydro,2-methyl	1.33	1128
1162		trans-p-2,8-menthadien-1-ol	traces	1133
1165	6711	1,2,3,5-tetramethylbenzene	0.75	1135
1182	3812	cyclohexane,pentyl-	0.43	1145
1187	53260	trans,cis-2,6-nonadien-1-ol	5.98	1149
1204	22819	2,6-nonadienal(E,Z)	2.56	1166
1207		camphor	traces	1162
1212	111411	2-nonenal	12.50	1166
1225	8590	1-nonanol	0.96	1175
1227	4178	octanoic acid	0.47	1177
1228		benzaldehyde,4-ethyl	traces	1177
1232	356	2-sec-butyl-3-methoxy pyrazine	0.04	1180
1252	4946	2-propanol,1-Butoxy	0.56	1194
1263	20025	2,4-nonadienal(E,E)	2.25	1203
1268		naphthalene	traces	1205
1274	2441	decanal	0.27	1209
1283		decane,2,5,6-trimethyl	traces	1215
1291	32225	2,4-nonadienal(E,E)	3.62	1221
1311	1994	β-cyclocitral	0.22	1235
1327	9336	3-decen5-one	1.05	1246
1333	2004	cis-4-decenal	0.23	1251
1353	61769	2-decenal(E)	6.93	1265
1357		nonanoic acid	traces	1268
1369	33255	terpenoid	3.73	1276
1383	8575	cycloheptene,5-ethylidene-1-methyl	0.96	1286
1395		terpenoid	traces	1294
1400	147991	2,4-decadienal(E,E)	16.61	1298
1413	307	6-propyl-m-cresol	0.04	1309
1421	1052	1-methylnaphthalene	0.12	1315
1431	112636	2,4-decadienal	12.64	1323
1438		3E,5Z-1,3,5-undecatriene	traces	1329
1442	1961	cis-4-decenal	0.22	1332
1448	3353	cyclopropane,nonyl-	0.38	1337

1466	6943	hydrocarbon(C-13)	0.78	1352
1471	1199	2-heptylfuran	0.14	1356
1475	1961	unknown compound,mw 208,bp 138	0.22	1359
1486	6340	trans-2-Undecenal	0.71	1368
1492	387	γ-decalactone	0.04	1373
1515	945	7-tetradecene	0.11	1392
1525	1737	tetradecane	0.19	1400
1593	5311	phenol,2,6-Bis(1,1-dimethylethyl)	0.59	1457
1614	5095	benzene sulfonic acid	0.572	1475
1622	2674	2-tert-BHA(anti-oxidant)	0.30	1482
1628	442	2,6-di-tert-butylbenzoquinone	0.05	1487
1644	497	pentadecane	0.06	1500
1649	644	β-Ionone	0.07	
1653	258	isocaryophyllene	0.03	
1675	1247	butylated hydroxy toluene(anti-oxidant)	0.14	
1692	217	5-pentylresorcinol	0.03	
1719	771	dihyrdroactinidiolide	0.09	
1726	293	thiophene,2-Butyl-5-(2-methylpropyl)	0.03	
1750	756	phenol,,4-(1,1-dimethylpropyl)	0.09	
1755	1482	2-heptyl furan	0.17	
1761	224	diethyl phthalate	0.03	
1820	not determined	euparone	traces	
1941	622	octyl salicylate	0.07	

5.4.4.5 Identification of volatiles and semi-volatile compounds in Sample GNG 12 deodorized Camelina Oil

A total of 154 compounds were identified in this sample and presented in Table 26. 2-Octenal (31.7ppm), 2, 4-decadienal (26.46 ppm),2, 4-heptadienal (27.5 ppm), 2-butenal (6.4 ppm), 2-pentenal (7.9 ppm),hexanal (7 ppm), 2-nonenal (10.3 ppm), 2-decenal (11.55 ppm),3,5-otcadien-3-one (8 ppm),nonanal (13.65 ppm),1-penten-3-ol (10.2 ppm),1-hepten-3-ol (7.14 ppm) dominated the headspace volatiles of this sample. Some saturated aldehydes from oxidation of oleic acid (20.27% from table 4) namely heptanal, octanal and decanal were present in small levels. 2-Pentyl furan was found highest in this sample and a similarity was observed with the other deodorized sample (GNG-13). These samples were subjected to only deodorization after extraction and filtration. These samples also had the highest level of oleic acid when compared to all other camelina oil samples used in this study (Table 4).

Pentanal, 2-ethylfuran, acetic acid,1-pentanol, 2-hexenal, 2,4-hexadienal, 2-butyl furan,heptanol,heptanoic acid, 2,6-nonadienal, 3,5-heptadien-2-one-6-methyl, 2-dodecenal were identified in small levels(1-3 ppm). Methyl vinyl ketone, γ-decalactone, β-farnesene, 2H-1-Benzopyran-2-one, β-ionone, 2-nonanone, n-hexanol, dimethyl sulfone, acetoin, 3-hexen-2-one,1-octen-3-ol were also identified in very small levels. Trace levels of cis-verbenol, 2-undecanone, farnesol, octanoic acid, p-cymene and limonene were also identified.

Appearance and aroma: Color of the oil was bright golden yellow and had an oily, nutty and mild green aroma.

Table 26: Volatile and semi-volatile compounds in deodorized camelina oil sample GNG-12 by Purge-and-trap headspace method

Scan #	Area Integration	Peak Assignment	Concentration PPM(w/w)	R.I.(Equity-5)
289	470	methyl vinyl ketone	0.06	591
295	699	2-butanone	0.08	595
303	302	hexane	0.04	600
305	co eluted	2,3-butanedione	traces	601
344	784	acetic acid	0.09	626
387	54570	2-butenal(E)	6.34	651
422	4957	3-buten-2-one,3-methyl	0.58	673
444	87521	1-penten-3-ol	10.17	686
447	co eluted	2-pentanone	traces	688
460	10962	pentanal	1.27	696
466	32391	heptane	3.76	700
469	7541	2-ethyl Furan	0.88	702
488		acetoin	traces	713
500	511	propanoic acid	0.06	727
541	14270	2-pentenal(E)	1.66	745
561	53807	2-pentenal isomer	6.25	757
573	33623	toluene-d8(Internal Standard)	3.91	764
581	51648	toluene	6.00	769
585	7457	cyclopentanecarboxaldehyde	0.87	771
589	20483	1-pentanol+2-hexenal(E)	2.38	773
610	4095	furan 2,3-dihydro-4-methyl	0.48	786
620	7642	1-octene	0.89	792
621	co-eluted with 620	butanoic acid	traces	792
634	114137	octane	13.26	800
641	60411	hexanal	7.02	804
648	15478	4-octene(E)	1.79	809
659	18172	2,4-octadiene	2.11	815
682	1113	1,3-octadiene	0.13	828
685	not determined	5-hexen-1-ol	traces	830
696	3923	3-hexene,2,5-dimethyl	0.46	837
703	2199	3-hexen-2-one	0.26	841
712	16395	2-hexenal	1.91	846
716	737	n-hexanol	0.09	849
725	19416	2-hexenal(E)	2.26	854
744	1492	p-xylene	0.17	865
752	524	hexyl alcohol	0.06	870
756	5343	m-xylene	0.62	872
769	2754	1,3,6-octatriene	0.32	880
786	3234	2-heptanone	0.38	890
792	2054	pentanoic acid	0.24	894
797	3138	o-xylene	0.37	897

803	21979	nonane	2.55	900
805	co eluted with 803	heptanal	traces	902
820	15910	2,4-hexadienal	1.85	911
832	540	heptane,2,4,6-trimethyl	0.06	919
841	1251	dimethyl sulfone+4-heptyn-3-ol	0.14	924
844	2621	2-hepten-1-ol	0.30	925
850	991	benzene,(1-methylethyl)-	0.12	929
855	118	3-cyclopentene-1,2-diol,cis	0.02	932
856	7886	octane,2,6-dimethyl	0.92	935
864	2239	cyclopentane,butyl	0.26	937
872	13385	2-butyl furan	1.56	943
883	not determined	2,5-dimethyl octane	traces	944
898	159608	2-heptenal(E)	18.54	959
910	10389	benzaldehyde +hydrocarbon (C-10)	1.21	966
912	traces	p-ethyl toluene	traces	967
920	3213	decane isomer	0.37	972
923		benzene,1,3,5-trimethyl	traces	974
931	18079	heptanol	2.10	979
939	3577	branched C-10 hydrocarbon	0.42	984
941	61490	1-hepten-3-ol	7.14	985
943	co eluted with 941	o-ethyl toluene	traces	987
945		cyclohexane,1-methyl-2-propyl	traces	988
951	not determined	2-octanone	traces	991
955	34142	2-pentyl furan	3.97	994
957		hexanoic acid	traces	995
965	146318	2,4-heptadienal(E,E)	16.99	1000
967	co eluted with 967	1,2,3-trimethyl benzene	traces	1001
976	34062	octanal	3.96	1007
990	91134	2,4-heptadienal isomer	10.59	1016
999	10397	1-methoxy-1,4-cyclohexadiene	1.21	1020
1003	8818	nonane,2,6-dimethyl	1.02	1025
1010	3068	p-cymene+benzyl alcohol	0.36	1029
1015	6256	p-mentha 1,3,8-triene	0.73	1033
1021	1135	limonene	0.13	1037
1033	26500	1-octen-3-ol	traces	1045
1037	co eluted with 1033	cycloheptanone,4-methyl-R	traces	1047
1046	10799	4-ethylcyclohexanol	1.26	1053
1055	6737	p-propyl toluene	0.78	1059
1060	129354	2-octenal	15.03	1062
1079	68907	3,5-octadien-2-one(E,E)	8.01	1075
1091	22114	heptanoicacid+unknown compound,bp 68,mw 154	2.57	1083
1100	6351	o- cymene	0.74	1089
1105	4339	2-nonanone	0.50	1092
1109	traces	2-ethyl,m-Xylene	traces	1095
1115	143647	trans-2-octenal	16.69	1099
1119	co-eluted with 1115	ethanone,1-(2,2 Dimethylcyclopentyl)	traces	1102

1128	117514	nonanal	13.65	1108
1142	not determined	3-ethyl,O-xylene	traces	1118
1152		octanoic acid methyl ester	traces	1126
1156	6165	2-hexyl furan	0.72	1129
1159	co eluted with 1156	trans-carveol	traces	1130
1178	3282	3-nonen-2-one	0.38	1143
1185	35078	trans,cis 2,6-nonadien-1-ol	4.07	1148
1191	47713	2-nonenal	5.54	1148
1201	23595	2,6-nonadienal(E,Z)	2.74	1159
1210	40981	2-nonenal(E)	4.76	1165
1215		para-diethylbenzene	traces	1168
1220	9531	3,5-heptadien-2-one,6-methyl,-(E)-	1.11	1172
1224	co eluted with 1220	para-ethylbenzaldehyde	traces	1175
1228		octanoic acid	traces	1178
1232	1700	1-nonen-4-ol	0.19	1181
1252	37794	diethyleneglycol butyl ether	4.39	1193
1266		naphthalene	traces	1204
1271	6538	decanal	0.76	1208
1288	31214	2,4-nondienal(E,E)	3.63	1220
1299	1555	2-decenal(Z)	0.18	1228
1308	1695	5-undecyne	0.19	1234
1319		unknown compound ,mw 135,bp 84	traces	1242
1326	33154	3-decen-5-one	3.85	1247
1328		2-decene,8-methyl-(Z)	traces	1248
1351	97928	2-decenal(E)	11.38	1265
1355	co eluted with 1351	decanol	traces	1267
1356	co eluted with 1351	nonanoic acid	traces	1268
1367	32809	terpenoid	3.81	1276
1372	1065	2-nonenenitrile	0.12	1280
1397	130376	2,4-decadienal(E,E)	15.15	1298
1402	38022	n-tridecane	4.42	1300
1417	1354	naphthalene,1-methyl	0.16	1313
1427	99037	2,4-decadienal(E,E)	11.51	1322
1434	23377	terpenoid	2.72	1327
1446	8026	cis-4-decenal	0.93	1337
1455	3122	1-dodecene	0.36	1345
1467	traces	2n-heptyl furan	traces	1355
1472	4474	unknown compound,bp 138,mw 208	0.52	1359
1484	33975	trans-2-undecenal	3.95	1369
1490	1886	γ-decalactone	0.22	1374
1495	3043	unknown compound,mw 160,bp 159	0.35	1378
1511	993	7-tetradecene	0.12	1391
1521	4543	2-undecenal+n-tetradecane	0.53	1400
1535	559	2-undecanone	0.07	1411
1590	5862	phenol,3,5-Bis(1,1-Dimethylethyl)	0.68	1458
1601	1230	beta-farnesene	0.14	1467
1607	4495	2H-1-benzopyran-2-one	0.52	1472

1614	21413	2-dodecenal(E)	2.49	1479
1619	1678	1,5-di-tert-butyl-3,3-dimethylbicyclo(3.1.0)hexan-2-one	0.19	1482
1624	974	2,5-di-tert-butyl-benzoquinone	0.11	1486
1638	1460	3-dodecen-1-yne(E)	0.17	1499
1640	1117	n-pentadecane	0.13	1500
1645	2210	β-ionone	0.26	
1658	2349	1-dodecanol	0.27	
1688		5-pentyl-resorcinol	traces	
1692	549	farnesol	0.06	
1716	1049	2(4H)-benzofuranone,5,6,7,7a-tetrahydro-4,4,7a-trimethyl(or dihydroactinidiolide)	0.12	
1726	426	3-isopropyl-4a,5-dimethyloctahydro-1(2H)-naphthalenone	0.05	
1746	2989	phenol,4-(1,1-dimethylpropyl)	0.35	
1752	4561	cis-verbenol	0.53	
1758	2462	diethyl phthalate	0.29	
1769	1172	2-propenal,(1,1-dimethylethyl)methyl-	0.14	
1816	1685	aristolone	0.19	
1950	1428	2-pentadecanone,6,10,14-trimethyl	0.17	
1998	1081	methyl palmitate	0.13	

5.4.4.6 Identification of volatiles and semi-volatile compounds in Sample GNG 13 deodorized Camelina Oil

A total of 171 compounds was identified in this sample and presented in table 27. The compounds 2-heptenal(19 ppm), 2,4-heptadienal (34 ppm), 2-octenal (37.93), 3,5-octadien-2-one (11.69 ppm), 2-nonenal (11.30 ppm), 2,4-nonadienal (6.5 ppm) and 2,4-decadienal (18.98) were found in high amounts indicating the auto-oxidation of linoleic and linolenic acids (22.1 and 21% respectively from Table 4). Compounds like nonanal (13 ppm), trans,cis,2,6-nonadien-1-ol (7.3 ppm), 2,6-nonadienal (3.4 ppm), 2-decenal (9.50 ppm), 4-decenal (9.4 ppm), 1-penten-3-ol (5.8 ppm) and 2-undecenal (3.6 ppm)were also found in high levels.

Compounds like 2-butenal, 3-pentanol-2-methyl, 1-hepten-3-ol, octanal, 3-octen-2-ol, 2-acetyl pyrrole, 1-decanol and 2-dodecenal were identified in moderate levels (2-6 ppm). Pentanal, 2-penten-1ol, 2-hexenal, 3-hexenal, 2-butyl furan, heptanol and 2-pentyl furan were also identified in small amounts (1-4 ppm). 2-Ethyl furan, 2-methyl furan, acetic acid,acetoin,isoamyl alcohol,toluene,3-hexen-1-ol,3-hexen-2-one, styrene, dimethyl sulfone,p-cymene,caryophllene,phenyl ethyl alcohol,β-ionone, isocaryphyllene, farnesol,dihydroactnidiolide and cis-verbenol were found in trace levels.

2-Heptenal, 2-pentylfuran, octanal, 2-octenal, 2-nonenal, trans, cis-2, 6-nonadien-1-ol, 2, 6-nonadienal and 2, 4-nonadienal were at high concentrations when compared to the other camelina oil samples in this study. 2-Sec-butyl-3-methoxy pyazine was identified at 0.08 ppm in this sample.**Appearance and aroma:** Color of the oil was golden yellow and had an oily, earthy and green bell-pepper kind of aroma.

Table 27: Volatiles and semi-volatile compounds in deodorized camelina oil sample GNG-13 by Purge-and-trap headspace method

Scan #	Area Integration	Peak Assignment	Concentration PPM(w/w)	R.I(Equity-5)
124	485	2-propenal	0.04	
134	623	propanal	0.05	
148	2151	**pentane**	0.19	500
180	146	2-pentene	0.02	520
197	255	1,3-pentadiene	0.02	531
263	127	1-propene-1-one,2-methyl	0.01	573
294	7648	2-butanone	0.68	592
306	1411	butanal+hexane	0.13	600
315	238	2-methyl furan	0.02	606
320	129	3-buten-2-ol	0.01	609
339	1137	ethyl acetate	0.10	620
369	1539	acetic acid	0.14	639
390	54120	2-butenal(E)	4.79	652
426	2999	3-buten-2-one,3-methyl	0.27	674
438	288	methyl acetate	0.03	681
447	traces	2-pentanone	traces	687
450	65775	1-penten-3-ol	5.83	688
465	16332	pentanal	1.45	698
469	22089	heptane	1.96	700
473	5934	2-ethyl furan	0.53	703
491	423	acetoin	0.04	713
496	2372	propionic acid	0.21	716
536	5637	isoamyl alcohol	0.49	740
545	86672	2-pentenal(E)	7.68	745
577	38378	toluene-D8(Internal Standard)	3.40	764
583	488	toluene	0.04	768
588	4948	cyclopentane carboxaldehyde	0.44	771
594	not determined	1-pentanol	traces	775
597	36906	2-peten-1-ol(E)	3.27	776
607		2-hexanol	traces	782
617	2785	furan,2,3-dihydro-4-methyl	0.247	788
622	6124	1-octene	0.54	791
635		butanoic acid	traces	797
637	81786	octane+hexanal	7.25	800
649	88202	3-pentanol,2-methyl	7.81	807
651	co eluted	4-Octene(E)	traces	809
655	4657	2-hexanol	0.41	811
662	12368	2,4-octadiene	1.09	815
664		3-octene(E)	traces	817
673	4952	1,4-heptadiene,3-methyl	0.44	824
681	4132	1,3-octadiene	0.37	827

701	7098	3-hexen-1-ol	0.63	840
707	5368	3-hexen-2-one	0.48	843
716	25746	2-hexenal	2.28	849
722	1062	2,3-pentanediol,3-methyl	0.09	853
728	19080	3-hexenal(Z)	1.69	856
733	5429	5-hexen-2-one	0.48	859
746	1429	nonane isomer	0.13	867
756	5755	hexyl alcohol	0.51	873
759	co eluted	p-xylene	traces	875
762	3582	2-hexenal(E)	0.32	877
771	4743	1,3,6-octatriene(E,E)	0.42	883
788	3002	2-heptanone	0.27	893
784		pentanoic acid	traces	894
795	2250	4-hexen-1-ol+styrene	0.20	898
797	4397	4-heptenal(E)+o-xylene	0.39	899
799	20173	nonane	1.78	900
807	co eluted	heptanal	traces	905
815	547	2-butoxy-1-ethanol	0.05	910
823	17067	2,4-hexadienal(E,E)	1.51	915
836		dimethyl sulfone	traces	922
840		2,5-dimethyl pyrazine+4-ethyl phenol	traces	924
846	6746	2-hepten-1-ol	0.59	929
851	3485	benzene,(1-methylethyl)-	0.31	931
863	5338	1,3,6-heptatriene,5-methyl(E)	0.47	939
867	3150	cyclopentane,butyl	0.28	940
876	38626	ethane,1-Methoxy-2-(methoxy methoxy)	3.42	946
882	12797	2-butyl furan+2-heptenal	1.13	950
888	3686	6-hepten-1-ol	0.33	954
903	211718	2-heptenal	18.76	963
904		tricyclo 4.2.0.02,4-oct-7-en-5-one	traces	963
913	54526	hydrocarbon(C-10)	4.83	969
915		benzaldehyde	traces	970
926	5000	1,3,5-trimethyl benzene	0.44	977
934	26735	heptanol+diisoamylene	2.37	981
945	88726	1-hepten-3-ol	7.86	988
954		2-octanone	traces	994
957	41468	2-pentyl furan	3.67	996
962	24233	hexanoic acid	2.15	998
967	220079	2,4-heptadienal(E,E)	19.49	1002
974		2-methoxy pyrazine	traces	1006
980	48473	octanal	4.29	1010
995	164637	2,4-heptadienal isomer	14.58	1020
1004	21923	6-octen-2-one	1.94	1026
1013	3996	benzyl alcohol	0.35	1031
1018	10023	p-cymene	0.89	1035
1024	3000	d-limonene	0.27	1040
1033	60276	3-octen-2-ol+2-octenal	5.34	1045

1040		cycloheptanone,4-methyl,R	traces	1050
1049	428197	2-octenal	37.95	1056
1053	co-eluted with 1053	2-octen-1-ol(E)		1059
1058	14072	C-11 hydrocarbon+ p-propyl toluene	1.25	1061
1070	1256	2-Hexen-1-ol acetate	0.111	1070
1073	83774	naphthalene,decahydro+2-acetyl pyrrole	7.42	1072
1082	1756	1-octanol	0.16	1078
1085	132039	3,5-octadien-2-one(E,E)	11.67	1078
1091	co eluted with 1085	heptanoic acid	traces	1084
1095	59857	4-isopropenylcyclohexanone	5.30	1087
1099	co eluted with 1095	3-ethyl-o-xylene	traces	1089
1105	13334	unknown compound,mw 138,bp 68		1093
1109	7487	2-nonanone	0.66	1096
1120	2561	ethanone,1-(2,2-dimethylcyclopentyl)	0.23	1103
1125	1543	isocamphane	0.14	1106
1132	143542	nonanal	12.72	1110
1153	1888	phenyl ethyl alcohol	0.17	1124
1156	not determined	Methyl octanoate	traces	1126
1161	8895	2-hexyl furan	0.79	1129
1166	13573	2-nonen-1-ol(E)	1.20	1133
1176	2379	1-nonen-3-ol	0.21	1139
1183	5372	3-nonen-2-one(E)	0.47	1144
1189	82166	trans,cis-2,6-nonadien-1-ol	7.28	1147
1195	127164	2-nonenal(E)	11.27	1151
1205	37898	2,6-nonadienal(E,Z)	3.36	1158
1226	30004	1-nonanol	2.66	1171
1229	not determined	benzaldehyde,4-Ethyl	traces	1173
1233	900	2-sec-butyl-3-methoxy pyrazine	0.08	1174
1234	9992	octanoic acid	0.89	1176
1254	28110	butyldiglycol+2-decanone	2.49	1190
1261	23557	2,4-nonadienal(E,E)	2.09	1194
1269	1566	naphthalene+alpha terpineol	0.13	1199
1274	7567	decanal	0.67	1203
1282	5471	2-decen-1-ol	0.48	1244
1291	50197	2,4-nonadienal isomer	4.44	1251
1307	1236	o-acetanisole	0.11	1262
1312	2880	2-heptenal,2-propyl	0.25	1266
1324	3608	3-decen-5-one	0.32	1276
1331	105963	cis-4-decenal	9.38	1282
1345	964	cyclodecanone	0.08	1293
1355	107267	2-decenal	9.50	1266
1359	50800	1-decanol	4.50	1269
1363		nonanoic acid	traces	1272
1370	32765	terpenoid	2.90	1278
1375		2-nonenenitrile	traces	1280
1384	9020	p-menth-4(8)en-9-ol	0.79	1284
1395	86271	tridecane	7.64	1300

1400	97083	2,4-decadienal(E,E)	8.60	1301
1406	1561	unknown compound ,mw 166,bp 123	0.14	1306
1421	1746	1-methylnaphthalene	0.15	1318
1432	117264	2,4-decadienal(E,E)	10.38	1326
1436		1,3,5-undecatriene(E,E)	traces	1330
1441	101675	tridecane isomer	9.00	1334
1474	2278	unknown compound,bp 138,mw 208	0.202	1360
1488	40223	trans-2-undecenal	3.56	1371
1494	1909	γ-decalactone	0.17	1376
1499	1142	trans-2-undcen-1-ol	0.10	1380
1505	1892	1-tridecene	0.17	1386
1515	4246	7-tetradecene	0.37	1393
1584	841	caryophyllene	0.07	1451
1593	4000	phenol,2,6-Bis(1,1-dimethylethyl)	0.35	1459
1600	3609	hydrocarbon(C-15)+2H(1)-benzopyran-2-one	0.32	1465
1617	27421	trans-2-dodecen-1-al	2.43	1479
1623	9002	2-tert-butyl-4-hydroxy anisole	0.79	1485
1628	1976	β-ionone	0.17	1489
1643	2906	pentadecane	0.26	1500
1653	1573	isocaryophyllene	0.14	
1686	400	trans-farnesol	0.035	
1720	1066	dihydroactinidiolide	0.09	
1750	4589	phenol,4-(1,1-dimethylpropyl)	0.41	
1757	10800	2,4-undecadienal(E,E)	0.97	
1760	co eluted	cis-verbenol	traces	
1765	1476	2-tert-butyldecahydronaphthalene	0.13	
1786	6375	2-tertbutylcyclohexanone	0.56	
1819	1744	aristolone	0.16	
1835	896	4-hexanoylresorcinol	0.08	

5.4.4.7 Identification of volatiles and semi-volatile compounds in Sample GNG 4 refined Camelina Oil

A total of 125 compounds were identified in this sample and the compounds are presented in table 28. The headspace volatiles was dominated by 2,4-heptadienal (26.1 ppm), 2-heptenal (6 ppm),1-penten-3-ol (9.6 ppm), octanal (4.7 ppm),2-octenal (7.88 ppm), 3,5octadien-2-one (5 ppm), nonanal (9.43 ppm), and 2,4-decadienal (18.02 ppm) which are some of the oxidation products of the unsaturated fatty acids (linolenic, linoleic and oleic acid). 2-butenal, 2-pentenal, 2-hexenal, 2, 4-hexadienal, heptanal, 2-butyl furan, 2-nonenal, decanal and 2-decenal were present in small to moderate levels. The oil had an alpha-linolenic acid content of 24.7% and linoleic and oleic acids content were 19.9 and 19.3% respectively (Table 4).

2-Pentylfuran, phenyl ethyl alcohol, 2, 6-nonadienal, acetone, 1-pentanol, 2-hexanol, 5-hexen-2-one, 2-heptanone, pentanoic acid were present in small levels (1-3 ppm). 2-Ethyl furan, propanal, 2-butanone, ethyl acetate, benzaldehyde, cis-verbenol, euparone, 2, 6-di-tert-butyl benzoquinone, 2-octen-1-ol and p-cymene were present in very small amounts.

Appearance and aroma: Color of the oil was pale yellow and had an oily, fatty and very mild leafy green aroma.

Table 28: Volatiles and semi-volatile compounds in refined camelina oil sample GNG-4 by Purge-and-trap headspace method

Scan #	Area Integration	Peak Assignment	Concentration PPM(w/w)	R.I(Equity-5)
124	569	2-propenal	0.06	
140	5188	pentane	0.52	500
141	co eluted	propanal	traces	501
161	956	2-pentene(E)	0.09	513
210	988	1,3-pentadiene(E)	0.09	544
292	2420	2-butanone	0.24	595
301	2017	hexane	0.20	600
333	739	ethyl acetate	0.07	620
345	1429	1,3-butadiene,2,3-dimethyl	0.14	628
357	8094	acetic acid	0.80	642
384	25564	2-butenal(E)	2.54	653
392	1827	3-methyl butanal	0.18	658
406	259	methyl acetate	0.03	667
420	995	3-buten-2-one,3-methyl	0.10	676
442	96439	1-penten-3-ol	9.60	690
445	co eluted with 442	2-pentanone	traces	692
458	31028	heptane+pentanal	3.09	700
466	3901	2-ethyl Furan	0.39	705
485		acetoin	traces	716
509	946	propanoic acid	0.09	733
529	412	3-penten-2-one(E)	0.04	742
538	10694	2-pentenal (E)	1.06	748
558	47173	2-pentenal isomer	4.69	758
571	37477	toluene-d8(Internal Standard)	3.73	767
576	3215	toluene	0.32	770
584	1903	1-pentanol	0.19	775
587	co eluted	2-penten-1-ol(E)	traces	777
617	1496	1-octene	0.15	794
618	co eluted	n-butyric acid	traces	795
634	106742	hexanal +octane	10.62	800
643	4150	4-octene(E)	0.41	809
656	3846	2,4-octadiene(E,E)+3-octene	0.38	816
665	3594	2-hexanol	0.36	821
692	6228	2-hexene,2,5-dimethyl	0.62	835
709	13430	2-hexenal(E)	1.34	845
724	co eluted	5-hexen-2-one	traces	853
766	3587	1,3,6-octatriene	0.36	876
783	1547	2-heptanone	0.15	885
785	co eluted with 783	pentanoic acid	traces	886
802	3997	heptanal	0.40	895
811		nonane	traces	900

817	3698	2,4-hexadienal(E,E)	0.37	904
837	1152	6,8-dioxabicyclo 3.2.1octane	0.11	917
861	782	diethylene glycol	0.08	932
870	12115	2-butyl furan	1.20	938
875	2511	2,3,3-trimethyl-1-hexene	0.25	941
886		bicyclo 3.3.1 nonane	traces	949
899	60069	2-heptenal(E)	5.98	957
906	1634	benzaldehyde	0.16	962
928	2501	heptanol+1-hepten-3-one	0.25	976
935	11309	1-hepten-3-ol	1.12	981
937	co eluted with 937	hexanoic acid	traces	983
940	2739	Branched C-10 hydrocarbon	0.27	985
942		7-octen-2-one	traces	985
947	7214	2-heptanone,6-methyl	0.72	989
952	8983	2-pentyl furan	0.89	991
964	141121	2,4-heptadienal(E,E)	14.04	999
970	47578	octanal	4.73	1003
975		propanol,1-(2-methoxypropoxy)(solvent)	traces	1007
988	121154	2,4-heptadienal isomer	12.05	1016
993	276	2-octen-1-ol	0.02	1021
1011	862	p-cymene	0.08	1027
1025	10194	cyclohexane,pentyl	1.01	1040
1037	79206	2-octenal(E)	7.88	1048
1041	4145	ccloheptanone,4-methyl-R	0.41	1051
1060	2676	2-(5-methylfuran-2-yl)-propionaldehyde	0.26	1064
1077	48282	3,5-octadiene-2-one(E,E)	4.80	1074
1085	8638	5,6-dihydro-2-pyranone	0.85	1081
1094	3390	cyclopentane,2-isopropyl-1,3-dimethyl	0.33	1086
1109	not determined	2-nonanone	traces	1096
1115	31020	undecane	3.08	1100
1126	94801	nonanal	9.43	1108
1129		4n-propyl resorcinol	traces	1110
1155	1194	2-hexylfuran	0.11	1128
1176	3903	3-nonen-2-one	0.38	1143
1183	11564	trans,cis-2,6-nonadien-1-ol	1.15	1148
1199	10460	2,6-nonadienal(E,Z)+isocamphane	1.04	1159
1202	co eluted with 1199	bicyclohexyl,2-ethyl-trans		1161
1207	9419	2-nonenal(E)	0.94	1164
1218	4601	3,5-heptadien-2-one,6-methyl-,-(E)	0.45	1172
1222	1566	2,6-dimethylbenzaldehyde	0.15	1175
1235	455	cyclononanone	0.04	1184
1245	14708	trans,4-nonenal	1.46	1191
1248	co eluted with 1245	butoxy ethoxy ethanol	traces	1193
1270	1627	decanal	0.16	1205
1285	6953	2,4-nonadienal(E,E)	0.69	1212
1307	883	2-decyn-1-ol	0.08	1223
1315	1206	4-tertbutylcyclohexene	0.12	1228

1322	7935	3-decen-5-one	0.79	1230
1336		naphthalene,decahydro-2-methyl	traces	1237
1349	30329	trans-2-decenal	3.02	1244
1364	15308	terpenoid	1.52	1251
1378	4969	cycloheptene,5-ethylidene-1-methyl	0.50	1258
1392	76504	terpenoid	7.61	1265
1427	181027	2,4-decadienal(E,E)	18.02	1283
1444	2697	cis-4-decenal	0.27	1291
1455	631	1-tridecene	0.06	1297
1461	6188	tridecane	0.62	1300
1465	5701	2-heptyl furan	0.56	1304
1471	2930	unknown compound,mw 208,bp 138	0.29	1311
1482	10743	trans,2-undecenal	1.07	1320
1500	1016	unknown compound,mw 160,bp 159	0.10	1337
1511	737	7-tetradecene	0.07	1349
1597	682	5-octyne-4-one,2,2,7,7-tetramethyl	0.07	1411
1610	2980	dodecanal	0.30	1422
1618	1778	2,4-diethoxy-1,5-formylbenzene	0.18	1430
1624	543	2,6,di-tert-butylbenzoquinone	0.05	1435
1649	452	1,3,7,7-tetramethyl-9-oxo-2-oxabicyclo(4.4.0)decane	0.05	1457
1658	1552	sesquiterpene	0.15	1465
1722	939	2-butyl-5-isobutylthiophene	0.09	
1746	2790	phenol,4(1,1-dimethylpropyl)	0.28	
1752	3613	2,4-undecadienal(E,E) + cis-verbenol	0.36	
1816	1757	euparone	0.17	

5.4.4.8 Identification of volatiles and semi-volatile compounds in sample GNG 22 refined camelina Oil

A total of 131 compounds were identified in this sample and the compounds are presented in Table 29. The headspace volatiles was dominated by 1-penten-3-ol (6.7 ppm), 2-pentanone (6.8 ppm), hexanal (7.20 ppm), 2-heptenal (9.7 ppm), 2,4-heptadienal (11 ppm), 2-octenal (11 ppm),nonanal (6.8 ppm), and 2,4-decadienal (11.07 ppm) which are some of the oxidation products of the polyunsaturated fatty acids. This sample had the highest level of α-linolenic acid (27.3% from Table 4) in this study and levels of linoleic, oleic and eicosenoic acids were 20.6, 19.25 and 14.21% respectively. 2-Penten-1-ol, 2-hexenal, 2-octanone, 2-pentyl furan and 3, 5-octadiene-2-one was present at levels ranging from 1 to 3 ppm.

Pentane, 2-ethyl furan, 2-propenal, 2-methyl propanal, 2-pentenal, 1-pentanol, 3-hexenal, 2-butyl furan, 2-decenal and 1-octen-3-ol were found in small amounts. Acetoin, 2-methoxy pyrazine and 2-hexyl furan were also identified in small levels. 3-Methyl butane, furfural, 2-heptanone, styrene, benzaldehyde, dimethyl sulfone, 2-octen-1-ol and p-cymene were present in trace amounts.

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Appearance and aroma: Color of the oil was mild to pale yellow and had an oily, nutty and very mild green aroma.

Table 29: Volatile and semi-volatile compounds in refined camelina oil sample GNG-22 by purge-and-trap headspace method

Scan #	Area Integration	Peak Assignment	Concentration PPM(w/w)	R.I.(Equity-5)
134	22750	2-propenal+2-propyn-1-ol	2.43	
148	29734	pentane	3.18	500
152		propanal	traces	502
168	1164	2-pentene(E)	0.12	512
198	2373	1,3-pentadiene(E)	0.25	534
266	229	2-propenal,2-methyl	0.02	573
277	176	1-propen-1one,2-methyl	0.02	579
289	9601	propanal,2-methyl	1.03	587
301	26277	2-butanone	2.81	594
310	39030	hexane	4.17	600
318		2-methyl furan	traces	605
342	2974	ethyl acetate	0.32	620
353	2007	2-pentene,3-methyl	0.21	627
386	27701	acetic acid	2.96	648
394	60410	2-butenal(E)	6.46	654
402		3-methyl butanal+2-butenal	traces	659
421	717	1-butanol	0.07	671
429	999	3-buten-2-one,3-methyl	0.11	676
438	565	2-buten-1-ol,2-methyl	0.06	682
451	62718	1-penten-3-ol	6.71	690
453	co-eluted	1-penten-3-one	traces	691
459	63770	2-pentanone	6.82	694
462	co-eluted with 459	3-butenoic acid	traces	695
467	64534	heptane+pentanal	6.91	700
475	26240	2-ethyl furan	2.81	705
494	4091	acetoin	0.44	716
509	7106	propionic acid	0.76	725
523	8332	isoamyl alcohol	0.89	733
538		2-methyl-1-butanol	traces	742
547	23071	2-pentenal(E)	2.46	747
551		2-hexanone	traces	749
556	2730	furan,2,3-dihydro,4-methyl	0.29	752
566	74093	2-pentenal(E)	7.92	758
579	34227	toluene-d8(Internal Standard)	3.66	766
585	4390	toluene	0.47	769
593	32990	1-pentanol+2-hexenal	3.53	774
596	36414	2-penten-1-ol(Z)	3.89	775
609	2999	2-hexanol	0.32	783
624	2223	1-octene+butanoic acid	0.24	792
638	49551	octane	5.30	800
642	67194	hexanal	7.19	802

649		4-octene(E)	traces	807
674	8164	2,4-octadiene	0.87	821
693	400	furfural	0.04	833
702	28121	3-hexenal(Z)	3.00	838
709	8504	3-hexen-2-one	0.91	842
730	41006	2-hexenal(E)	4.38	854
748	230	ethyl benzene	0.03	865
760	653	n-hexanol	0.07	872
773	4370	1,3-Trans,5-cis-octatriene	0.46	880
790	1077	2-heptanone	0.12	890
795		pentanoic acid	traces	893
797	2665	4-hexen-1-ol+styrene	0.28	894
809	9700	heptanal	1.04	901
824	5947	2,4-hexadienal(E,E)	0.64	911
830	8019	3-penten-1-ol,4-methyl	0.86	914
843	2046	dimethyl sulfone	0.22	921
874	1174	ethane,2-(2-methoxyethoxy)-(solvent)	0.13	942
877	13605	2-butyl furan	1.45	944
882	10198	2-heptyn-1-ol	1.09	947
893	2079	1-hepten-3-ol	0.22	954
899	90579	2-heptenal(E)	9.68	959
909	6189	trans-4,4-dimethyl-2-hexene	0.66	964
914	559	benzaldehyde	0.06	967
924	4194	1-heptanol	0.45	973
934	5400	1-octene,6-Methyl	0.58	980
942	34141	2-octanone	3.65	985
945	co-eluted with 945	7-octen-4-ol	traces	987
954		2-heptanone,4-methyl	traces	992
958	32898	2-pentyl Furan	3.52	995
959		pentanoic acid,3-methyl	traces	995
968	95700	2,4-heptadienal(E,E)	10.23	1001
973	co-eluted with 973	1-methoxy,1,4-cyclohexadiene	traces	1004
980	19540	octanal	2.09	1009
1006	7817	2,4-heptadienal isomer	0.84	1026
1015	910	benzyl alcohol	0.09	1032
1019	1287	p -cymene	0.14	1035
1026	112	d-limonene	0.02	1039
1039	1131	cycloheptanone,4-methyl	0.12	1048
1049	10472	1-octen-3-ol+2-nonanone	1.12	1054
1060	86070	2-octenal(E)	9.20	1063
1064	49837	undecane	5.33	1064
1067	co-eluted with 1064	2-hexen-1-ol,acetate(E)	traces	1066
1069		2-acetyl pyrrole	traces	1068
1078		1-octanol	traces	1074
1084	38939	3,5-octadien-2-one(E,E)	4.16	1077
1085	co-eluted with 1084	heptanoic acid	traces	1078
1093	15765	4-isopropenyl cyclohexanone	1.69	1083

1102	4991	3-oxatricyclo 5.2.0(2,4)nonan-8-one	0.53	1090
1132	63731	nonanal	6.82	1109
1133	co-eluted with 1133	3-nonen-1-ol(E)	traces	1110
1138		4n-propyl resorcinol	traces	1113
1147	164	6-methyl-2-pyrazinyl methanol	0.02	1119
1162	2927	2-hexyl furan	0.31	1128
1170	1433	p-ethyl styrene	0.15	1134
1182		3-nonen-2-one	traces	1142
1188	8548	trans,cis-2,6-nonadien-1-ol	0.91	1146
1193	15710	2-nonenal(E)	1.68	1149
1204	8793	2,6-nonadienal(E,Z)	0.94	1156
1219		octanoic acid	traces	1166
1223		3,5-heptadien-2-one,6-methyl(E)	traces	1168
1228	1449	p-ethyl benzaldehyde	0.15	1172
1240	472	1-nonen-4-ol	0.05	1179
1253	5947	ethanol,2-(2-butoxyethoxy)	0.64	1188
1270	4732	2,4-nonadienal	0.51	1199
1274	1917	decanal	0.21	1201
1327	2974	3-decen-5-one+2-decenal(E)	0.32	1228
1341	854	2-furanacetaldehyde,a-isopropyl	0.10	1235
1354	27460	2-decenal	2.94	1242
1356	co-eluted	nonanoic acid	traces	1243
1369	12779	terpenoid	1.37	1250
1383	2351	cycoheptene,5-ethylidene-1-methyl	0.25	1257
1396		terpenoid	traces	1264
1401	51059	2,4-decadienal(E,E)	5.46	1267
1407	1449	3,4-heptadiene,3,5-diethyl	0.15	1270
1432	52528	2,4-decadienal isomer	5.61	1283
1436	20285	terpenoid	2.17	1284
1449	1049	cis-4-decenal	0.11	1291
1466	1962	tridecane	0.21	1300
1471		2-heptyl furan	traces	1306
1475	2268	unknown compound,mw 208,bp 138	0.24	1311
1487	7432	2-undecenal	0.79	1326
1623	2331	3-BHA(antioxidant)	0.25	
1629	293	2,6-di tertbutylbenzoquinone	0.03	

6. CONCLUSIONS

6.1 Fatty acid Composition

A total of 21 different fatty acids were identified in 18 camelina oil samples (unrefined, deodorized and completely refined) using GC-FID and GC-MS methods. The 5 major fatty acids observed were α-linolenic, linoleic, oleic, eicosenoic and palmitic and these accounted for 80-85% of the fatty acids identified in camelina oil. The other 16 fatty acids were found at varying levels in small quantities and 3 fatty acids, namely tricosanoic (C23:0), pentadecanoic (C15:0) and heptadecanoic (C17:0) are being reported here for the first time. Erucic acid (C22:1, n-9) was found in all samples and and presence of eicosenoic acid (C20:1, n-9) makes the fatty acid profile of camelina oil unique when compared to other edible oils like canola, olive, soybean, sunflower and safflower.

Refined camelina oil sample GNG 22 was the best source of α-linolenic acid in the sample set characterized for fatty acids and the levels of α-linolenic acid observed in this study were lower than published reports in Europe and the United States.

The unsaturated fatty acid content of camelina oil and the ratio of linolenic to linoleic acids make it a nutritionally favorable vegetable oil. Camelina oil can be used for a number of applications including cooking oil, ω-3 enriched food products, nutraceutical and pharmaceutical products once it gets the FDA-GRAS status.

6.2 Unsaponifiables fraction

The Unsaponifiables fractions were extracted using the AOAC method 28.081 (William Hortwiz, 1980) and the individual components were identified using GC-MS. Eight camelina oil samples were analyzed and the unsaponifiables fraction ranged from 0.450.80%.

Unrefined camelina oil sample GNG 17 was observed with the highest content of unsaponifiables in this study.

The unsaponifiables fraction identified in camelina oil consisted mainly of free sterols and di- and tri-terpene compounds. Beta, gamma, delta and alpha tocopherols were identified in very small amounts in some samples. Unsaponifiable compounds in major concentrations were β-sitosterol, campesterol, squalene, cholesterol, phytol and brassicasterol and these compounds contributed to 80-90% of the total unsaponifiables content.14-methylergost-8-en-3-ol, γ-sitosterol, obtusifoliol, stigmasterol, δ5-avenosterol and cycloartenol were found in smaller amounts.

6.3 Free fatty acids

The free fatty acids in eight camelina oil samples were determined by AOAC method 28.029. The free fatty acids in unrefined and deodorized camelina oil samples were found between 0.50-0.80 %(expressed as oleic acid). The free fatty acids in refined camelina oil samples ranged between 0.03-0.06 %(as oleic acid). The FFA values of unrefined, deodorized and refined camelina oil samples were below the limit(less than 1 % and 0.1%) recommended in the vegetable oil industry (Belitz and Grosch, 1999).

6.4 Volatiles and semi-volatiles

Volatiles and semi-volatile compounds (168) in camelina seed samples were identified using both static and purge-and-trap headspace methodologies. Lipid oxidation products, nitrogen containing compounds, sulfur compounds and minor levels of maillard reaction products contributed to the aroma of camelina seeds and meal. The maximum number of volatile compounds was identified in sample # GNG-Omega meal and the naturally occurring pyrazines (2-sec butyl-3-methoxy pyrazine and 2-isopropyl-3-methoxy pyrazine) were identified in 3 samples: Ligena 1 pre-heated & crushed sample, Ligena 1 crushed seed sample and GNG-omega meal sample. The compound allyl isothiocyanate, responsible for the typical mustard aroma was found only in 1 sample # GNG-omega meal.

Volatiles and semi-volatile compounds in camelina oil samples were identified using purge-and-trap method headspace method. A total of 306 volatile and semi-volatile compounds were identified in 8 camelina oil samples and some of these compounds were identified in camelina seeds and meal also. Lipid oxidation products of unsaturated fatty acids namely acids, alcohols, ketones, aldehydes, furans and hydrocarbons dominated the headspace volatiles. Phenolic compounds, quinones, nitrogen containing heterocyclic compounds, sulfur compound, aromatic benzene compounds and terpenes were also identified in varying levels.

The aroma of unrefined camelina oil samples were oily, earthy, green (bell-pepper kind) and mild sulfury. Refined oil samples had a mild aroma with oily, green notes and the aroma of the deodorized samples were mild oily and green (bell-pepper type).

6.5 Summary

Camelina oil's unique aroma was probably due to the presence of several compounds contributing to green notes (leaf alcohols, aldehydes and 2-pentyl furan), 2-sec-butyl-3-methoxy pyrazine and sulfur compounds in oils. The compound 2-sec-butyl-3-methoxy pyrazine was present at concentrations (0.04 to 0.08 ppm) higher than its odor detection threshold (0.001 ppm) and might be responsible for the earthy, green bell - pepper kind of aroma of unrefined camelina oil samples. This compound was not detected in refined camelina oils suggesting that it could have been removed during the refining process. Presence of sulfur containing compounds (1-butene-4-isothiocyanato, 2, 4, 5-trithiahexane), lipid oxidation and minor levels of maillard reaction products contribute to mustard, oily and green notes to camelina oil.

Camelina seeds contained sulfur compounds (carbonyl sulfide, methyl mercaptan, dimethyl sulfide, dimethyl di- & trisulfide, 2, 4, 5-trithiahexane, allyl isothiocyanate and 1-butene-4-isothiocyanato) which can impart mustard and cabbage like odors. Pyrazine compounds like 2-isopropyl-3-methoxy pyrazine and 2-sec-butyl-3-methoxy pyrazine can contribute to earthy, green vegetable-like notes were also identified in some seed samples and can be perceived as off-odors and affect the aroma and flavor profile of camelina meal.

The fatty acid profile, unsaponifiables fraction and volatiles and semi-volatiles study on camelina oil can be helpful in understanding the influence of geographical conditions, oil refining methodologies and cultivar variety on the chemical quality of the oil produced in Montana.

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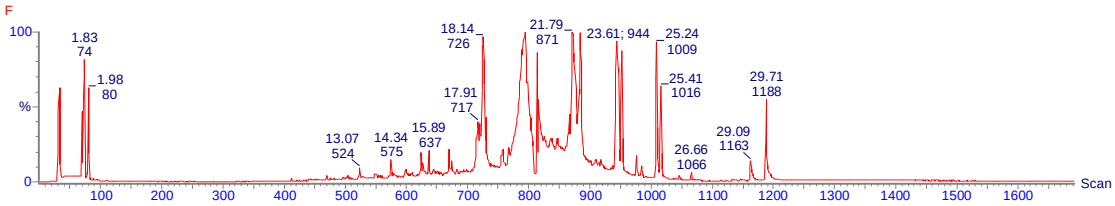
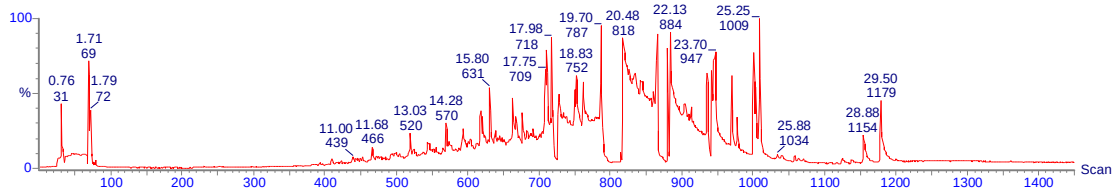
8. APPENDIX

8.1 Fatty acid Methyl esters

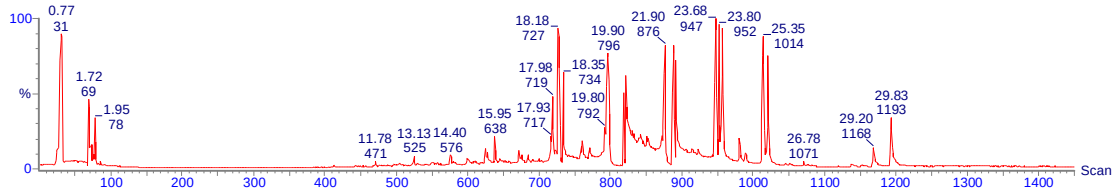
1. Total ion chromatogram of esterified sample GNG-1C107, unrefined camelina oil by GC-MS



2. Total ion chromatogram of esterified sample GNG-9, unrefined camelina oil by GC-MS

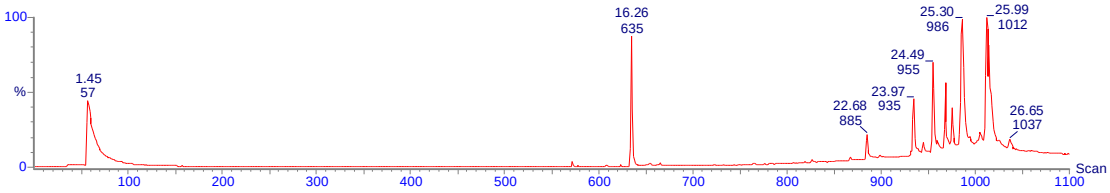


3. Total ion chromatogram of esterified sample GNG-13, deodorized camelina oil by GC-MS

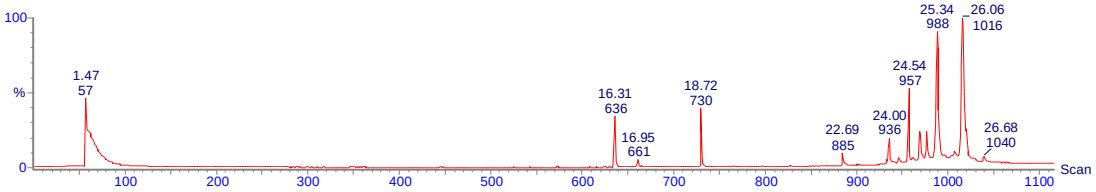


8.2 Unsaponifiables

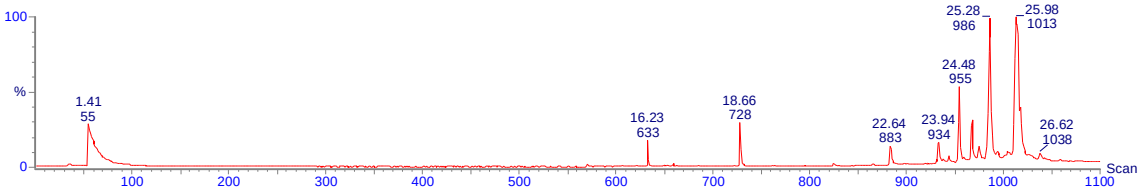
1. Total ion chromatogram of Unsaponifiables fraction of sample GNG 17 MK 1206 unrefined camelina oil by GC-MS



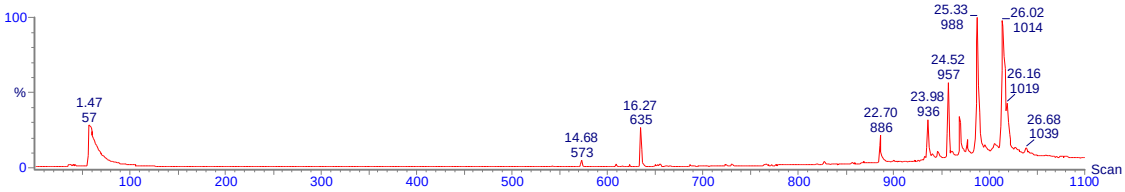
2. Total ion chromatogram of Unsaponifiables fraction of sample 18 M 1206 unrefined camelina oil by GC-MS



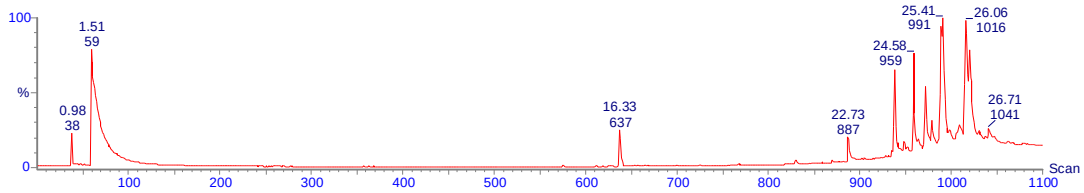
3. Total ion chromatogram of Unsaponifiables fraction of sample GNG-19-C107-unrefined camelina oil by GC-MS



4. Total ion chromatogram of Unsaponifiables fraction of sample GNG-1C-107 unrefined camelina oil by GC-MS

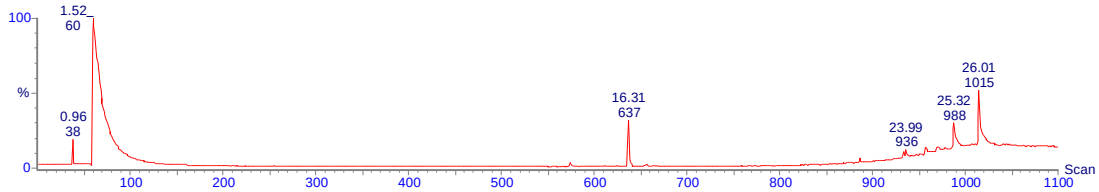


5. Total ion chromatogram of Unsaponifiables fraction of sample GNG-4 RBD refined camelina oil sample by GC-MS

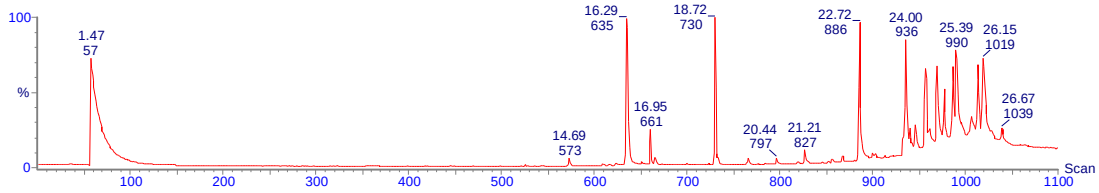


6. Total ion chromatogram of Unsaponifiables fraction of sample GNG-22 RBD 107 refined camelina oil sample by GC-MS

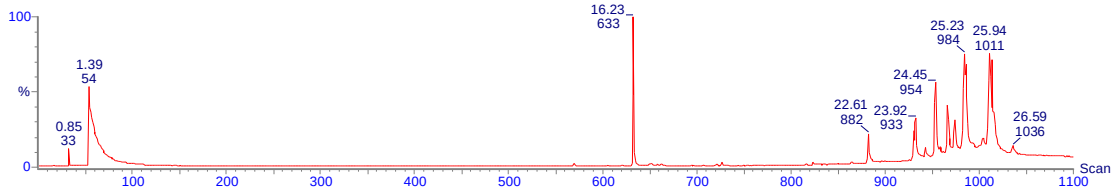
G



7. Total ion chromatogram of Unsaponifiables fraction of sample GNG-12 deodorized camelina oil by GC-MS

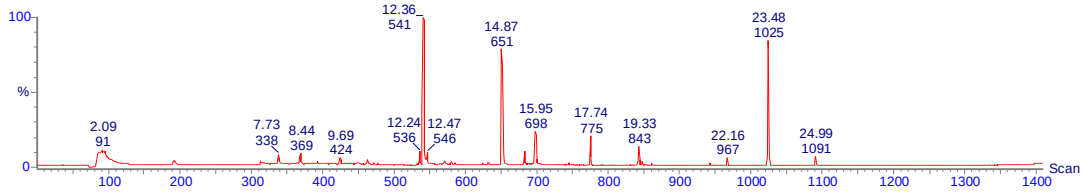


8. Total ion chromatogram of Unsaponifiables fraction of sample GNG-13 deodorized camelina oil by GC-MS

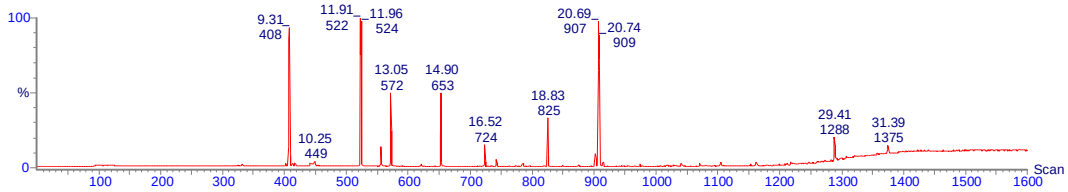


8.3 Volatiles and semi-volatiles

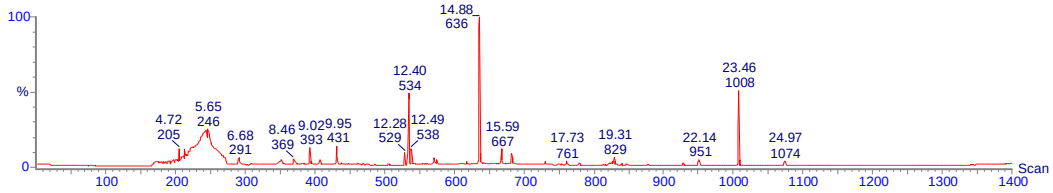
1. Total ion chromatogram of volatiles in sample Ligena 1 camelina seeds by HDPSC-GC-MS



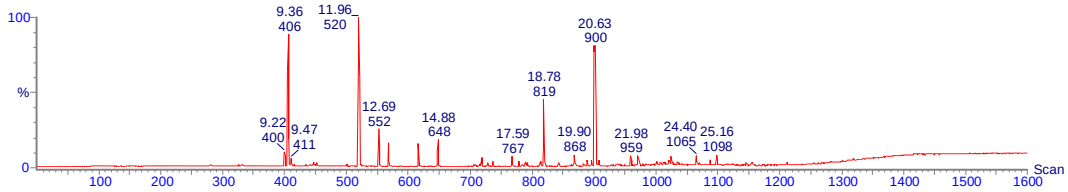
2. Total ion chromatogram of volatiles and semi-volatiles in sample Ligena 1 camelina seeds by P&T-TD-GC-MS



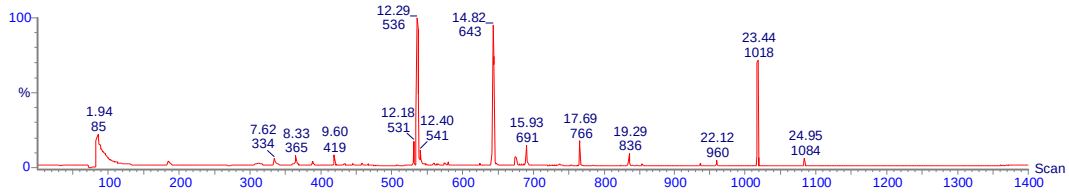
3. Total ion chromatogram of volatiles in sample Ligena 1A camelina seeds by HDSPC-GC-MS



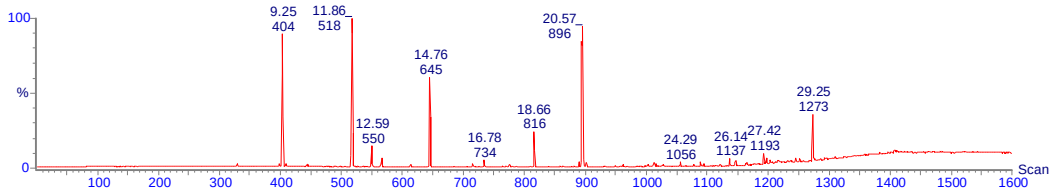
4. Total ion chromatogram of volatiles and semi-volatiles in sample Ligena 1A camelina seeds by P&T-TD-GC-MS



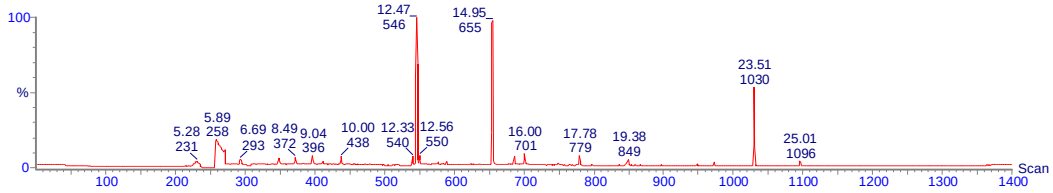
5. Total ion chromatogram of volatiles in sample Calena 2 camelina seed by HDSPC-GC-MS



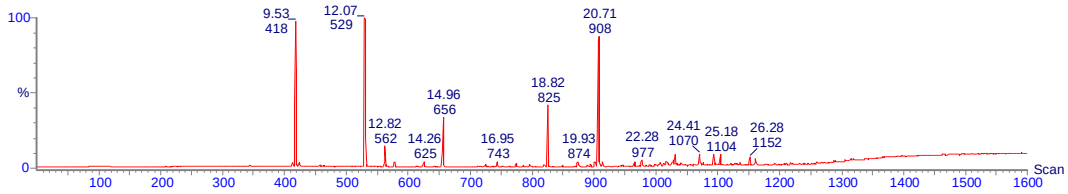
6. Total ion chromatogram of volatiles and semi-volatiles in sample Calena 2 seeds by P&T-TD-GC-MS



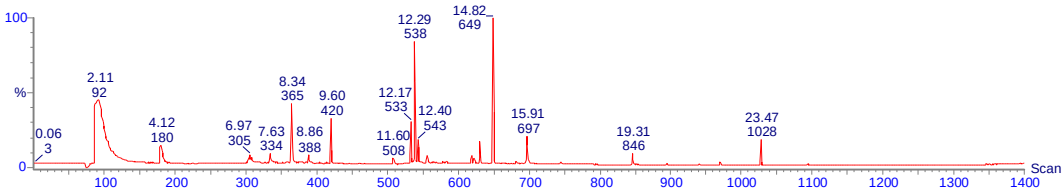
7. Total ion chromatogram of volatiles in sample Calena 2A camelina seed samples by HDSPC-GC-MS



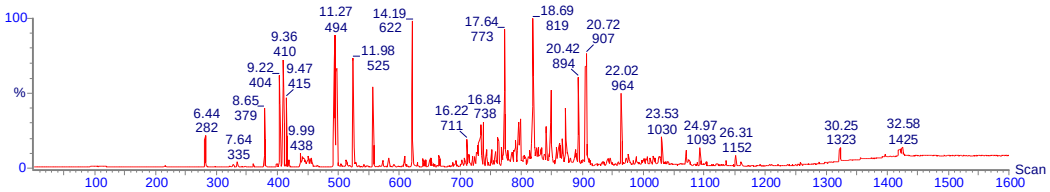
8. Total ion chromatogram of volatiles and semi-volatiles in sample Calena 2A camelina seeds by P&T-TD-GC-MS



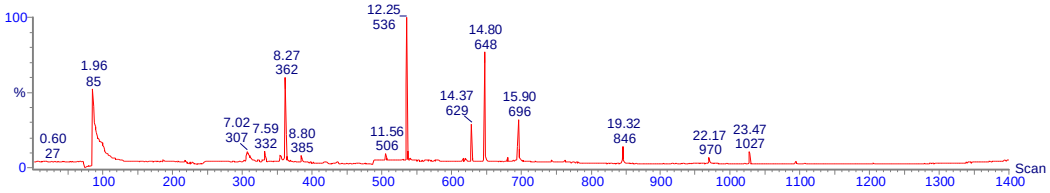
9. Total ion chromatogram of volatiles in sample GNG-omega meal by HDSPC-GC-MS



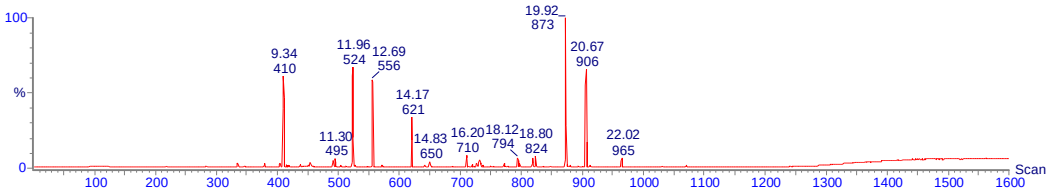
10. Total ion chromatogram of volatiles and semi-volatiles in sample GNG-omega meal by P&T-TD-GC-MS



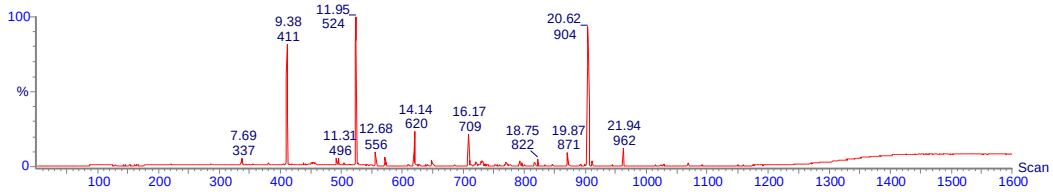
11. Total ion chromatogram of volatiles in sample Ligena 1 crushed seeds by HDSPC-GC-MS



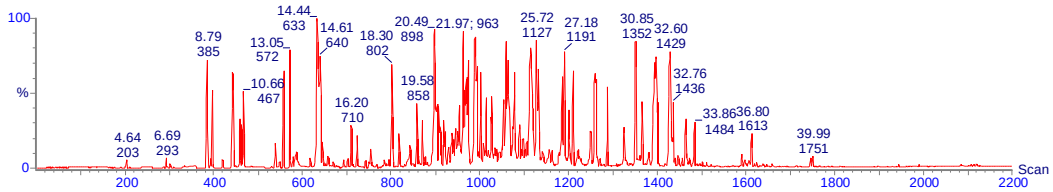
12. Total ion chromatogram of volatiles and semi-volatiles in sample Ligena 1 crushed seeds by P&T-TD-GC-MS



13. Total ion chromatogram of volatiles & semi-volatiles in sample Ligena 1-preheated & crushed seeds by P&T-TD-GC-MS

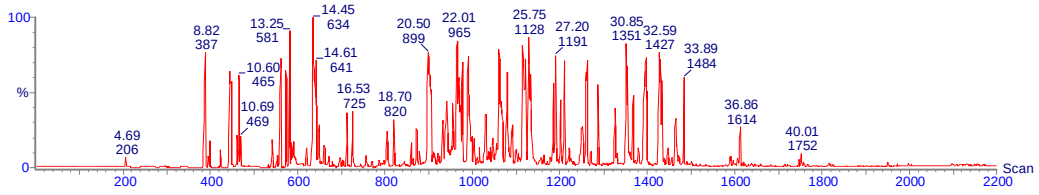


14. Total ion chromatogram of volatiles and semi-volatiles in sample GNG-17 MK 1206 camelina oil by P&T-TD-GC-MS

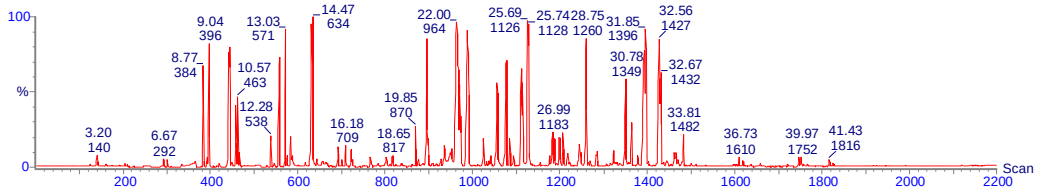


15. Total ion chromatogram of volatiles and semi-volatiles in sample GNG-12 deodorized camelina oil by P&T-TD-GC-MS

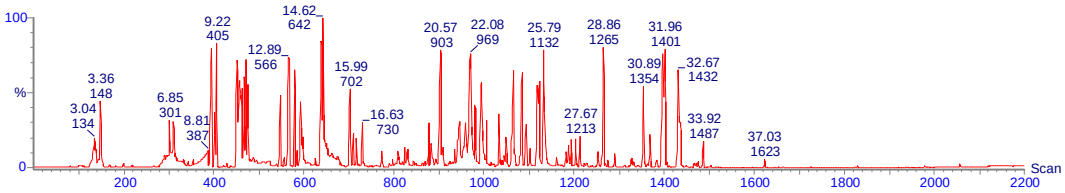
G



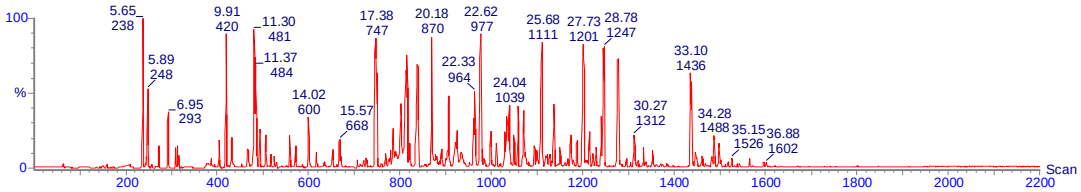
16. Total ion chromatogram of volatiles and semi-volatiles in sample GNG-4 RBD 107 refined camelina oil by P&T-TD-GC-MS



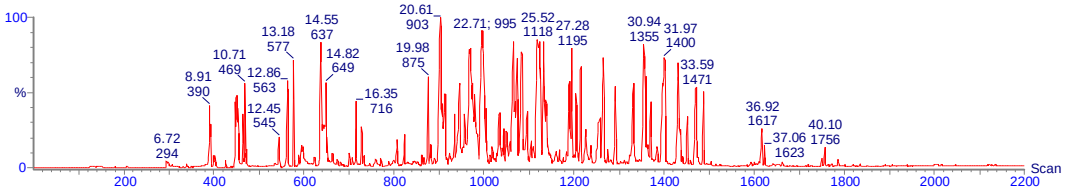
17. Total ion chromatogram of volatiles and semi-volatiles in sample GNG-22 RBD 107 refined camelina oil by P&T-TD-GC-MS



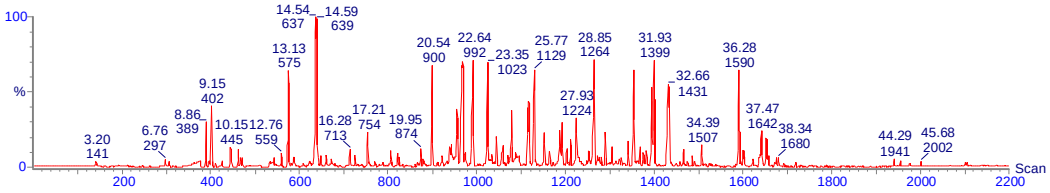
18. Total ion chromatogram of volatiles and semi-volatiles in sample GNG-19 C 107 unrefined camelina oil by P&T-TD-GC-MS



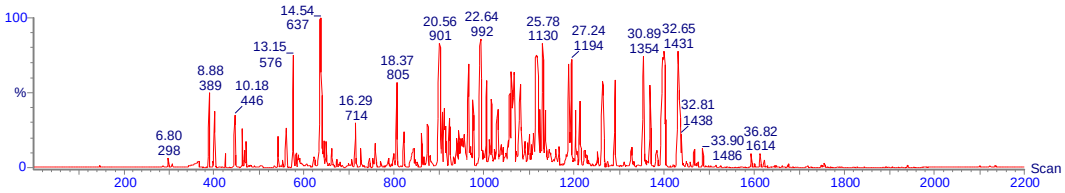
19. Total ion chromatogram of volatiles and semi-volatiles in sample GNG-13 deodorized camelina oil sample by P&T-TD-GC-MS



20. Total ion chromatogram of volatiles and semi-volatiles in sample GNG-1C 107 camelina oil by P&T-TD-GC-MS



21. Total ion chromatogram of volatiles and semi-volatiles in sample GNG-18 M 1207 unrefined camelina oil by P&T-GC-MS



Curriculum Vita

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Education

09/2005 Masters Program, Rutgers University, New Brunswick, NJ

06/1997 M.Sc, Food technology, CFTRI (Central Food technological Research Institute) CFTRI, Mysore, India.

07/1995 B.Sc (Physics), Justice Basheer Ahmed Sayeed Women's College, Chennai, India.

Work Experience

09/06-05/09 Graduate student, Mass-spectrometry lab

07/98-07/03 Creative Flavorist, BBA/IFF India Ltd., St.thomas Mount, Chennai, India.

07/97-07/98 Analytical officer,R&D,Britannia Industries Ltd.,Padi,Chennai,India.