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Research Article

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The Characterization of Volatile Compounds of Lupin Türkiye Genotype HS-SPME/GC-MS Method

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HIGHLIGHTS

- Volatile Compounds of Lupin Türkiye Genotype
- HS-SPME / GC-MS Method
- 65 µm PDMS/DVB fiber is the most preferable in terms of compound identification, with 97 compound.

Abstract

The aim of this study is to reveal the volatile and semi-volatile constituent of the local *Lupinus albus* L. genotype grown in Türkiye by using the HS-SPME technique. Local lupin is a legume plant with great potential due to its high seed yield and protein and oil content in the seeds. Powdered seeds of local lupin genotypes were analysed and compared with respect to types and contents of volatile semi-volatile compounds contents using four different SPME fiber with GC-MS system in W9N11, SWGDR4G4 and SWGDR4G5 libraries. Fiber with 50/30 µm DVB/CAR/PDMS identified 54 compounds, 65 µm PDMS/DVB fiber 97 compounds, 85 µm Carboxen/PDMS fiber 28 compounds, and 85 µm polyacrylate 12 compounds. As a result, 65 µm PDMS/DVB fiber is the most preferable in terms of compound identification, with 97 compound. In terms of content, Benzene, Methyl(1-Methylethyl) (39.92%), Gamma-Terpinene (12.26%), Cis-Ocimene (5.93%), 1,3,6-Octatriene,3,7-Dimethyl-(% 5.51), Beta-Myrcene (4.70%), Alpha-Pinene (4.39%), Alpha.-Thujene (4.21%), Alpha Terpinene (2.55%), Camphene (2.23%), 1,6-Octadien-3-Ol,3,7-Dimethyl (2.10%) were mostly detected in the analysis with 50/30 µm DVB/CAR/PDMS fiber. With the other fiber 65 µm PDMS/DVB; phospine oxide, triphenyl- (15.49 %), pulegone (5.03 %), L-linalool (3.85 %), cyclopentasiloxane, decamethyl (3.54 %), cyclohexasiloxane, dodecamethyl (3.13 %), cyclotrisiloxane, hexamethyl (2.32 %), oxime-,methoxy-phenyl (2.08 %), 1-pentanamine,N-pentyl (1.60 %), 3,5-octadıen-2-one (1.55 %), I-menthone (1.50 %). Fiber 85 µm Carboxen/PDMS; the most four components in the content are; Benzene, 2, 4-diisocyanato-1-methyl (54.27 %), Pyrrole-3-carbonitrile, 5-formyl-2, 4 (18.41 %), 1-Butanol, 4-(1-methylethoxy) (14.67 %), pyridine,4-(1-pyrrolidinyl)- (8.13 %). With 85 µm polyacrylate fibre the most found component is 1, 2-Benzenedicarboxylic acid, diethyl ester (98.90 %). In conclusion, as far as we know this study is the first study about the volatile content of local lupin genotype of Türkiye.

Keywords: Volatile compounds, Solid phase microextraction, Lupin, Local genotype, HS-SPME, GC-MS.

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1. Introduction

Lupinus is a large and diverse genus that belongs to the Fabaceae family and includes about 170 species (Gresta, 2017). *Lupinus albus* "white lupin" has a wide distribution in the Mediterranean region. It is planted over all the Mediterranean region and also in Egypt, Syria, Sudan, Ethiopia, Central and Western Europe, South America and USA, Tropical and Southern Africa, Ukraine, and Russia. Like legume seeds, lupin contains high amounts of protein, minerals and dietary fibre. The protein content of white lupin seed (33-47%) is higher than other legumes and close to the soy protein content (Dervas et al., 1999).

Lupins have a long date of for using both as ornamental plants in gardens and as an agricultural crop. There are four lupin species *L. albus*, L. *angustifolius*, *L. luteus* and *L. mutabilis* that have gained agricultural significance. Lupin seeds and flour are used in several cereal products such as pasta, crisp, bread, cookie, cake and breakfast cereal (Dervas et al., 1999; Erbaş et al., 2005; Yaver and Bilgiçli, 2021). Lupin seeds have admitted increasing international interest as an alternative source of human food ingredients due to their high-quality protein and dietary fibre. The definition of seed content is very important for breeding and crossing studies. According to the literature, headspace solid phase microextraction (HS-SPME) and gas chromatography-mass spectrometry (GC-MS) has gained wide approval as effective extraction technique for various samples in the last twenty years (Cuevas-Glory et al., 2007; Panighel and Flamini, 2014; Xu et al., 2016; Royandazagh ans Pehlivan, 2016). Solid phase microextraction (SPME) involves the adsorption of analytes upon a fused silica fibre coated with proper stationary phases and their following desorption instantly before chromatographic analysis (Arthur and Pawliszyn, 1990; Bicchi et al., 2000; Kataoka et al., 2000; Krutz et al., 2003; Pawliszyn, 2012; Ulrich, 2000; Zhang and Pawliszyn, 1993). The target analytes can be adsorbed on the fibre by immersing it in the sample or by exposing it to the sample headspace (HS-SPME), in which case matrix interferences can be highly reduced. This properly reduces analysis time and improves least detection limits, while maintaining resolution and can be used in two main modes, that is direct-extraction and headspace configurations. Due to these benefits, HS-SPME is strongly used to sample the volatile components from aromatic and medicinal plants (Bicchi et al., 2004; Muselli et al., 2009; Ercan and Dogru, 2022). Simsek Sezer et al. (2023) was determined to volatile compounds belonging to some lupin genotypes by SPME GC-MS.

The aim of this study is to reveal the volatile and semi-volatile constituent of the local *Lupinus albus* variety grown in Türkiye by using the HS-SPME technique.

2. Materials and Methods

Lupinus albus seeds grown under Destigin/Doğanhisar-Konya/Türkiye conditions were used as material. Seeds are local population grown in that region.

2.1. Seed Propagation

Local lupin genotypes were studied to identify volatile and semi-volatile content during our analyses. Seeds are stored in the department of Field Crops of Agriculture Faculty at Selçuk University. Seeds were dried at ambient temperature without sunlight exposure. Dried seeds were ground by using a hand grinder. The ground samples (3gr) were diluted and sealed in a 10 ml vial. The HS-SPME fibres, 50/30 μm DVB/CAR/PDMS, 65 μm PDMS/DVB, 85 μm Carboxen/PDMS and 85 μm polyacrylate were preferred for analysis. The SPME apparatus was directly injected into the upper space of the vial to adsorb volatile compounds and then directly injected into the Shimadzu QP2010ULTRA GC-MS apparatus using a Restek Rxi-5 MS capillary column.

2.2. *Analyses of GC-MS*

The volatile compounds of lupin were analysed by applying the method of (the injector temperature was 250 °C) using SPME-GC-MS. Compounds were isolated by a 15 min. SPME fibre exposure into a GC injector at 250 °C. The extracts from the SPME procedure were analysed on a Shimadzu QP2010 ULTRA FID GC–MS system. A 30 m length Restek Rxi-5 MS column (0.25 mm id, film thickness 0.25 μm) was used. Carrier gas was helium with a flow rate as 1.8 mL/min. The GC oven temperature was programmed to hold at 40 °C for 3 min and then to increase to 240 °C at 5 °C/min, finally holding at 240 °C for 3 min. The detector ion source temperature was 200 °C, and the interface temperature was set at 250 °C. Mass spectra were acquired in the electron impact mode at 70 eV, using m/z range of 50–350 and 2 s scan time.

2.3. *Data Analyses*

Chromatograms of SPME fibers all samples were subjected to noise reduction prior to peak area integration, and later, the peak areas of components in the chromatogram were integrated (Figure 1). Compounds were identified by comparing with three libraries, W9N11, SWGDR4G4 and SWGDR4G5. Compounds mostly matched in W9N11 library. Identification of components in the sample was based on the retention time (RT). The identification of the components present in the samples was calculated using Kovats retention index. The relative rate of the volatile oil compounds was obtained from peak areas. All analyses were performed in three replications, and all the numeric data are means of three independent analyses.

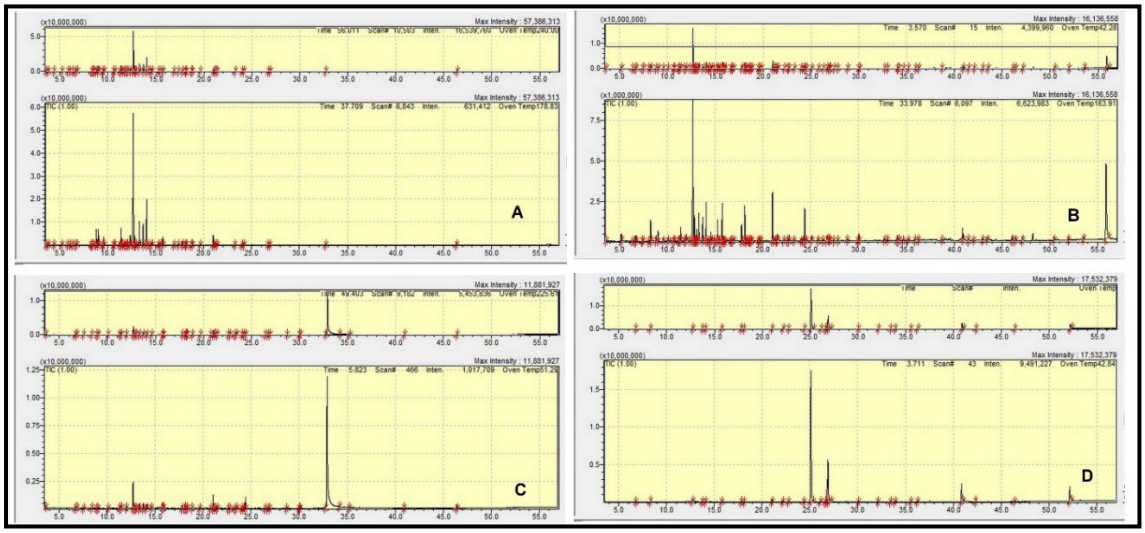

Figure 1. The total ion chromotograms of studied local lupin specimens via different SPME fibers (A: 50/30 μm DVB/CAR/PDMS, B: 65 μm PDMS/DVB, C: 85 μm Carboxen/PDMS and D: 85 μm polyacrylate)

3. Results

This section may be divided by subheadings. It should provide a concise and precise description of the experimental results, their interpretation, as well as the experimental conclusions that can be drawn.

Totally 147 compounds were separated and identified from the studied local lupin sample with 50/30 μm DVB/CAR/PDMS, 65 μm PDMS/DVB, 85 μm Carboxen/PDMS and 85 μm polyacrylate fibres respectively (Table 1). The total ion chromatograms (TIC) of studied local Lupin specimens were given Fig.1. With 50/30 μm DVB/CAR/PDMS fiber; the twelve components that are the most found in content; benzene methyl (1-methylethyl) (39.92 %), gamma-terpinene (12.26 %), cis-ocimene (5.93 %), 1,3,6-Octatriene,3,7-Dimethyl (5.51 %), beta-myrcene (4.70 %), alpha-pinene (4.39 %), alpha-thujene (4.21 %), D-Limonene (2.86 %), Pulegone (2.69 %), alpha terpinene (2.55 %), camphene (2.23 %), 1,6-octadien -3-ol,3,7-dimethyl (2.10 %). With the other fiber 65 μm PDMS/DVB; benzene methyl (1-methylethyl) (23.61 %), phospine oxide, triphenyl- (15.49 %), pulegone (5.03 %), L-linalool (3.85 %) and gamma-terpinene (3.79 %), cyclopentasiloxane, decamethyl (3.54 %), cyclohexasiloxane, dodecamethyl (3.13 %), cis-ocimene (2.58 %), D-Limonene (2.39 %), cyclotrisiloxane,

hexamethyl (2.32 %), 1,3,6-octatriene,3,7-dimethyl (2.24 %), oxime-,methoxy-phenyl (2.08 %), 1-pentanamine,N-pentyl (1.60 %), 3,5-octadıen-2-one (1.55 %), I-menthone 1.50 %). Fiber 85 µm Carboxen/PDMS; the most four components in the content are; Benzene, 2, 4-diisocyanato-1-methyl (54.27 %), Pyrrole-3-carbonitrile, 5-formyl-2, 4 (18.41 %), 1-Butanol, 4-(1-methylethoxy) (14.67 %), pyridine,4-(1-pyrrolidinyl)- (8.13 %). With 85 µm polyacrylate fibre the most found component is 1, 2-Benzenedicarboxylic acid, diethyl ester (98.90 %) (Figure 2).

In accordance with analyses, four compounds are common for all fibres with variable proportions. These compounds are benzene methyl (1-Methylethyl), gamma-terpinene, cyclopentasiloxane, decamethyl and pulegone respectively.

Table 1. The % peak area values of identified compounds of studied samples

Compound Name	% Area			
	50/30 µm DVB/CAR/PDMS	65 µm PDMS/DVB	85 µm Carboxen/PDMS	85 µm Polyacrylate
1-Butanol,3-Methyl-	-	0.13	-	-
Cyclotrisiloxane,Hexamethyl-	0.17	0.76	-	-
Benzene,Ethyl-	0.05	0.09	-	-
O-Xylene	-	0.54	-	-
1-Hexanol	0.39	0.52	-	-
Styrene	-	0.09	-	-
Benzene,1,2-Dimethyl-	-	0.24	-	-
Ethanol,2-Butoxy	-	0.05	-	-
Oxime-,Methoxy-Phenyl-	0.11	**2.08**	**-**	**-**
Propanamide,3-Amino-3-(Hydroxyimino)	-	0.43	-	-
Alpha-Pınene,	**4.39**	0.96	**-**	**-**
Bicyclo(2.1.1)Heptane,2,2-Dimethyl-3	-	0.23	-	-
Benzaldehyde	-	0.25	-	-
Chloroacetic Acid,Heptyl Ester	-	0.12	-	-
Bıcyclo(3.1.1)Heptane ,6,6-Dımethylethyl	-	0.15	-	-
Hexanoic Acid	-	0.11	-	-
3-Octanone	0.70	0.52	-	-
Beta-Myrcene	**4.70**	1.30	**-**	**-**
Decane	-	0.15	-	-
Cyclotetrasiloxane,Octamethyl-	0.15	0.74	0.04	-
Alpha-Terpinyl 3-Methylbutanoate	-	0.16	-	-
Benzene,Methyl(1-Methylethyl)-	**39.92**	**23.61**	0.62	0.25
D-Limonene	**2.86**	**2.39**	**-**	**-**
1,8-Cineole	0.72	0.26	-	-
1-Hexanol,2-Ethyl-	-	0.26	-	-
Benzyl Alcohol	-	1.15	-	-
Cis-Ocimene	**5.93**	2.58	**-**	**-**
1,3,6-Octatriene,3,7-Dimethyl-	**5.51**	2.24	0.05	
Gamma-Terpinene	**12.26**	3.79	0.08	0.04
Nonane,5-(2-Methylpropyl)-	-	0.30	-	-
Cıs-Sabınene Hydrate	0.07	0.17	-	-
3,5-Octadıen-2-One	0.63	**1.55**	**-**	**-**
3-Oxatricyclo(4.1.10(2.4))Octane,2,7,7	-	0.10	-	-
Benzene(2-Methyl-2-Propenyl)-	-	0.32	-	-
Cyclotrisiloxane,Hexamethyl-	**-**	**2.32**	**-**	**-**
L-Linalool	**-**	**3.85**	0.33	0.05
3-Ethyl-3-Methylheptane	-	0.48	-	-
Nonanal	-	0.40	-	0.03
Undecane,5-Methyl-	-	0.22	-	-
Benzaldehyde,2,5-Bis((Trimethylsily)Oxy)	-	0.42	-	-
Octanoic Acid,Methyl Ester	-	0.12	-	-
Limonene Oxide	-	0.19	-	-
I-Menthone	**-**	**1.50**	0.11	**-**
Cyclopentasiloxane,Decamethyl-	0.10	**3.54**	0.07	0.08
P-Menthone	-	0.45	-	-
1-Borneol	-	0.32	-	-
1-Nonanol	-	0.11	-	-
Isopulegone	-	0.17	-	-
Terpinen-4-Ol	-	0.10	-	-

Table 1. (Continue)

Naphthalene	0.08	0.40	-	-
Benzene,1-Methoxy-4-(2-Propenyl)-	-	0.15	-	-
Dodecane	0.07	0.29	-	-
Decanal	-	0.12	-	-
Pulegone	2.69	**5.03**	0.64	0.15
Benzene,1-Methoxy-4-Methyl-2-(1-Methyletyhl)	-	0.38	-	-
2,5-Cyclohexadiene-1,4-Dione,2-Methyl-	-	0.19	-	-
Benzaldehyde,4-Methoxy-	-	0.69	-	-
Lactose	-	0.15	-	-
Hexane,3,3-Dimethyl-	-	0.06	-	-
Anethole	-	0.64	-	-
Tridecane	0.05	0.23	-	-
Dodecane,2,2,11,11-Tetramethyl-	0.15	0.21	-	-
Tridecane,3-Methyl	-	0.27	-	-
Cyclohexasiloxane,Dodecamethyl-	**-**	**3.13**	0.11	0.12
3-Hexanol,2-Methyl-5-Nitro-,(R@,R@)-	-	0.26	-	-
Benzene,2,4-Diisocyanato-1-Methyl-	**-**	0.88	**54.27**	**-**
Propanoic Acid,2-Methyl-,3-Hydroxy-2,4	-	0.21	-	-
Cyclopentasiloxane,Decamethyl-	-	0.14	-	-
Tetradecane	-	0.44	-	-
Beta-Clovene	-	0.17	-	-
Undecanal	-	0.05	-	-
Hexasiloxane,Tetradecamethyl-	-	0.09	-	-
Beta-Copaene	-	0.06	-	-
1-Butanol,4-(1-Methylethoxy)-	**-**	0.17	**14.67**	**-**
Pentadecane	-	0.10	-	-
Cycloheptasiloxane,Tetradecamethyl-	-	0.69	0.05	
A Tetradecanol	0.14	0.15	-	-
Hexadecane	-	0.13	-	-
4,4,5,6-Tetramethyltetrahydro-1,3-Oxazin	-	0.52	-	-
Phenol,2,6-Bis(1,1-Dimethlethyl)-4-	-	0.16	-	-
Trans Methyl Dıhydrojasmonat	-	0.10	-	-
Cyclooctasiloxane,Hexadecamethyl-	-	0.20	-	-
Octadecane	-	0.11	-	-
Benzoic Acid,2-Ethylhexyl Ester	-	0.26	-	-
Nonadecane	-	0.09	-	-
1-Pentanamine,N-Pentyl-	**-**	**1.60**	**-**	**-**
Hexadecanyldimethylamine	-	0.13	-	-
Hexadecanoic Acid, Methyl Ester	-	0.14	-	-
1,2-Benzenedicarboxylic Acid,Dibutyl Ester	-	0.24	-	-
Eıcosamethylcyclodecasıloxan	-	0.42	-	-
Morpholine,4-Octadecyl-	-	0.46	-	-
Dodecanoic Acid,İsooctyl Ester	-	0.55	0.15	-
Cyclooctasiloxane,Hexadecamethyl-	-	0.50	-	-
Cyclononasiloxane,Octadecamethyl-	-	0.39	-	-
Bis(Di(Trimethylsiloxy)Phenylsiloxy)Trimethyl	-	0.34	-	-
1H-Purin-6-Amine((-2-Fluorophenyl	-	0.30	-	-
Phosphine Oxide,Triphenyl-	**-**	**15.49**	**-**	**-**
1-Pentanol (Cas)	0.31	-	-	-
Butanoic Acid,2- Methyl-Methyl Ester	0.55	-	-	-
Octane	0.07	-	-	-
2- Butenoic Acid, 3- Methyl-, Methyl Ester	0.04	-	-	-
Butanoic Acid,2- Methyl- Ethyl Ester	0.06	-	-	-
Benzene,1,4-Dimethyl-	0.34	-	-	-
(-)-Beta-Pinene	0.09	-	-	-
Tricyclene	0.12	-	-	-
Alpha-Thujene	**4.21**	**-**	**-**	**-**
Bicyclo (2.1.0)Hex-2-Ene,4-Methylene-1-	0.11	-	-	-
Camphene	**2.23**	**-**	**-**	**-**
Sabinene	0.20	-	-	-
Beta-Pinene	0.75	-	-	-
Ethyl Amyl Carbınol	0.21	-	-	-
Alpha-Phellandrene	0.48	-	-	-
Delta.3 Carene	0.41	-	-	-
Alpha Terpinene	**2.55**	**-**	**-**	**-**
Cis-Ocimene	**5.93**	**-**	**-**	**-**
Cyclohexene, 1- Methyl-4-(1-Methylethyl)	0.28	-	-	-
1,6-Octadien -3-Ol,3,7-Dimethyl	**2.10**	**-**	**-**	**-**
Alloocimene	0.38	-	-	-
2,4,6- Octatriene,3,4-Dimethyl-	0.25	-	-	-
Cyclohexanone,5- Methyl- 2-(1-Methylethyl)	0.86	-	-	-
Cyclohexanone, 5- Methyl-2-(1-Methyl)	0.23	-	-	-
Cıs- Isopulegone	0.04	-	-	-
Dodecane	0.09	-	-	-
Carvacrol Methyl Ether	0.19	-	-	-
2,5- Cyclohexadiene-1,4-Dione,2-Methyl	0.19	-	-	-
Tetradecane,2,2-Dimethyl-	0.16	-	-	-
Longifolene	0.07	-	-	-

Table 1. (Continue)

Laurınsaeure , 4- Octylester	0.57	-	-	0.05
Formic Acid,Hexyl Ester	-	-	0.03	-
4-Ethylbenzoic Acid,Cyclopentyl Ester	-	-	0.06	-
Beta-Ocimene	-	-	0.06	-
2-Ethoxyethyl Acrylate	-	-	0.61	-
Benzene,1-Methoxy-4-(2-Propenyl)-	-	-	0.12	-
1,3-Benzenediamine,4-Methyl-	-	-	0.24	-
7-Amino-1,3-Dihydro-İndol-2-One	-	-	0.26	-
Pyridine,4-(1-Pyrrolidinyl)-	**-**	**-**	**8.13**	**-**
Pyrrole-3-Carbonitrile,5-Formyl-2,4	**-**	**-**	**18.41**	**-**
3H-Imidazo(4,5-F)Quinoline,2-Amino-3	-	-	0.11	-
1,4-Dıbutoxybutane	-	-	0.08	-
Benzophenone	-	-	0.21	-
3H-Imidazo(4,5-F)Quinoline,2-Amino-3	-	-	0.06	-
1(2H)-Quinolinecarboxylic Acid,6-Amino	-	-	0.32	-
Formic Acid,2-Methylhex-3-Yl-Ester	-	-	0.19	-
Trans Beta-Ocimene	-	-	-	0.04
1,2-Benzenedicarboxylic Acid,Diethyl Ester	**-**	**-**	**-**	**98.90**
7,9-Di-Tert-Butyl-1-Oxaspirp(4,5)Deca-6,9	-	-	-	0.15
2-Ethylhexyl Methyl İsophthalate	-	-	-	0.05
The number of identified compound	**54**	**97**	**28**	**12**

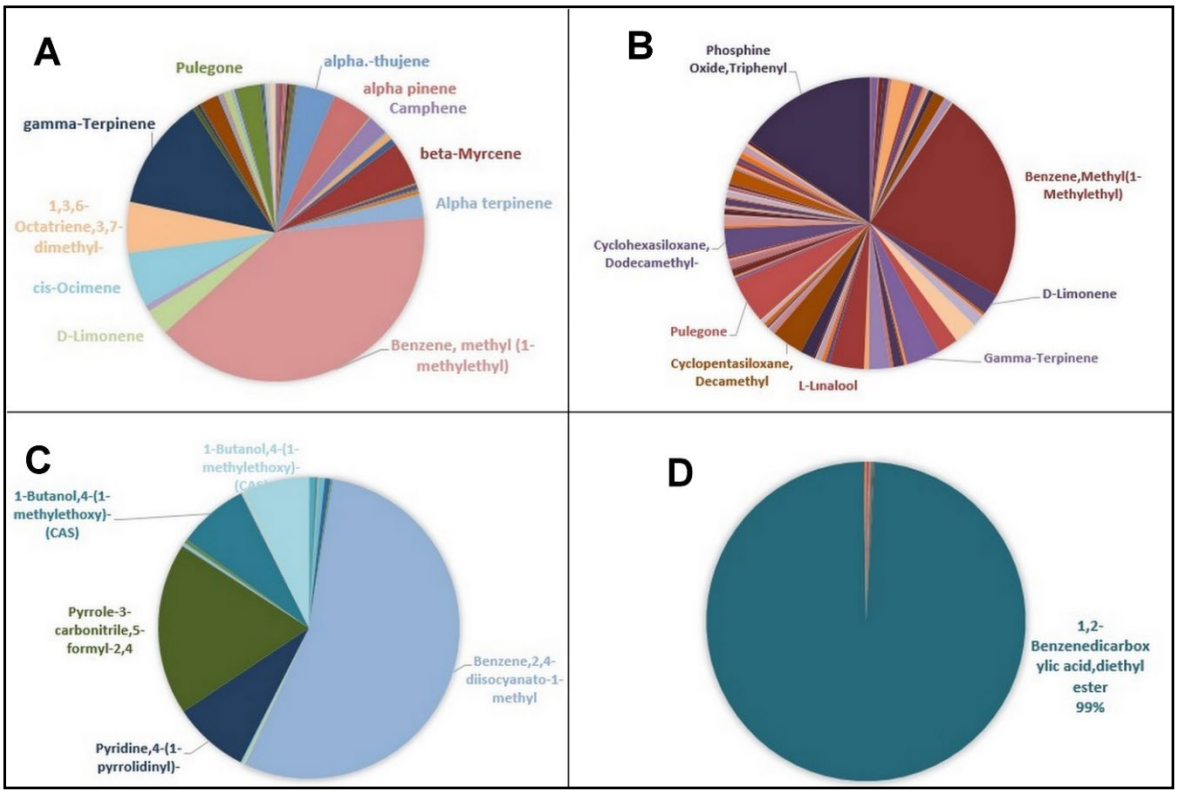

Figure 2. The pie charts of most found compounds in studied samples

4. Discussion

As far as we know, there are limited studies to reveal the content and the volatile composition of local lupin seeds. Some of the work on this subject is inadequate. The phenolic compound profiles and antioxidant capacities of wild and varieties *L. albus* L. seeds were determined before. The total phenolic content (TPC), antioxidant activity, radical scavenging activity and ferric-reducing antioxidant power (FRAP) in a beta-carotene-linoleic acid emulsion have been detected (Karamać et al., 2018). Also, the nutritional and chemical properties of white lupin (*L. albus* L.) were characterized via the HPLC system (Erbaş et al., 2005). Straková et al. (2006) the nutritional composition of the Lupinus genus seeds was reported in the study. They compared Lupin seeds and soybean and reported Lupin seeds significantly exceeded the content of crude protein in

soybeans. In the other study, the chemical composition of a new Lupin species from Spain; Lupinus mariae-josephi has been revealed. The chemical composition of this species is found similar to other lupin species in terms of total protein, oil, and alkaloid contents . Liquid chromatography-mass spectrometry was used to identify phenolic compounds (Múzquiz et al., 2011). Some physical properties and nutritional compositions of lupin (*L. albus* L.) seeds in Türkiye were reported (Yorgancılar et al., 2018). Similarly, the mineral content of debittered white lupin (*L. albus* L. local genotypes) seeds was determined (Yorgancilar et al., 2009). In addition, the nutritional and chemical changes of bitter and sweet lupin (*L. albus* L.) bulgur products were determined Yorgancilar and Bilgiçli (2014). In another study conducted in local lupin seeds (*L. albus* L.) the metabolite content was determined and the mite and insecticidal effects of the seed extract were investigated (Elma et al., 2021).

5. Conclusions

In this study, local lupin seeds from the Konya region were analyzed using four different fibers via SPME-GC/MS. According to the analysis with SPME fibers, we can clearly say that PDMS/DVB is the most preferable fiber in terms of compound identification. As far as we know this study is the first study about the volatile composition of local lupin genotype from Türkiye and characterization of seed content is very important for breeding and crossing studies.

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Conflicts of Interest: The authors declare no conflict of interest.

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