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Abstract

BACKGROUND: *Humulus lupulus* L. is a dioecious herbaceous perennial plant with a climbing habit, whose female inflorescences, commonly known as cones, produce and accumulate bitter substances and essential oils. The present study aimed to assess the adaptability of some American hop varieties (Cascade, Chinook, Comet) in the Mediterranean environment of the Calabria region (Italy) through the evaluation of the morpho-biological and productive characteristics and the characterization of the aromatic traits of the inflorescences.

RESULTS: Cascade emerged as the earliest variety. Different morphological traits were ascertained among the studied varieties. Comet proved to be the most productive variety, with a dry cone production of 0.35 t ha^{-1}, while chinook the earliest. Essential oil was obtained by hydrodistillation and analysed by a combination of GC-FID and GC-MS. Myrcene, β-caryophyllene and α-humulene were the main components.

CONCLUSION: The combination of leaf and cone dimensions could be adopted as useful tools together with the determination of the aromatic profiles to discriminate varieties. From the agronomic point of view Comet was the most productive variety, while Chinook emerged as the earliest one; concerning the essential oils Comet and Chinook showed similar profiles, Comet was different especially for the sesquiterpenes content.

Keywords: Humulus lupulus, morphological traits, cone yields, essential oi

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INTRODUCTION

Hop plant belongs to the genus *Humulus* comprising three different species: *H. lupulus*, *H. japonicus* and *H. yunnanensis*[1]. The genus belongs to the *Cannabaceae* family, which was included in the *Rosales* order since 2003.[2] Plants show consistent differences in distinct parts of the world, therefore several subspecies have been identified.[1]

Humulus lupulus L. is a dioecious herbaceous perennial plant with a climbing habit, whose female inflorescences, commonly known as cones or strobiles, produce and accumulate bitter substances and essential oils.[3] Its unique bittering and flavouring properties have led to the plant becoming a widely cultivated crop,[4] mainly in Germany and the USA.[2] As well as α- and β-acids contribute to the bitterness of the beer balancing the sweetness of the malt, volatile compounds give the beer its characteristic hoppy aroma.[5] Different chemical substances contribute in various and complementary ways to developing aromatic traits in beer: aldehydes give grassy flavours, esters give citrusy flavours, etc.[6] Each cultivar has a characteristic α-, β-acids and essential oils profile, but variations are expected due to the specific environmental conditions.[7] Indeed, factors affecting hop yield and quality characteristics include temperatures and soil water content;[8] in particular, accumulation of resins is inhibited by high temperatures.[7] In a study carried out in the Czech Republic, Mozny et al.[9] found that an increase in air temperature leads to changes in the timing of the phenological stages which are earlier onset, resulting in shorter intervals between them.

The crop requires areas with moderate temperatures and rainfalls, conditions that are generally found between 45° and 50° latitude.[10] Long and warm days are needed to ensure a successful flowering and thus a suitable cones yield, as well as winter temperatures of approximately 4 °C or less are necessary to satisfy the cold requirements of the crop, which is variety dependent, for a period of one to two months.[11] Although the utilisation of hop strobiles is almost entirely related to the brewing industry,[2] the earliest uses were for culinary (young shoots) and medicinal purposes[12] due to their counteracting effects on microbes and viruses.[13] In the modern brewing industry, hop has become a crucial element for the characterization of brewing products. As reported by Van Holle et al.,[14] hop brewing quality and value is associated both with cultivation area and variety. Indeed, there is a growing interest in the impact that the growing area may exert on the biochemical composition of the female inflorescences. The term terroir, which can be defined as the set of biotic and abiotic factors related to environment influencing the crop traits in a certain zone,[15] commonly related to wine, cocoa, coffee, is therefore gaining interest in the hop industry.[14]

Hop plant is spontaneous in many environments of Southern Italy, anyway it has never been extensively cultivated in this area, and this is probably due to a less beer consumption, strong competition with grape widely cultivated in all Italy and high irrigation requirement of hop.[16] However, nowadays the situation has changed and hop cultivations are growing everywhere both for research purposes and for short-chain brewing industry. As a consequence of the development of the craft beer movement worldwide, the cultivation of hops has also spread to new regions far from the traditional areas. The same happened in Italy, where there is still a lack of knowledge and experience in the management of this crop in new environments.[16] The craft beer sector has also established itself in Italy as one of the most enterprising and constantly developing phenomena, despite dependence on foreign countries for the supply of raw materials, including hops. Since 2016, the area involved in hop cultivation in Italy has increased by 80 hectares, with the prevalence of small farms.[17] With this in mind, the present study aimed to assess the adaptability of three American hop varieties (Cascade, Chinook, Comet) in the Mediterranean environment of the Calabria region (Italy) through the evaluation of the morpho-biological and productive characteristics and the characterization of the aromatic traits of the inflorescences.

MATERIALS AND METHODS

Plant material and experimental design

The experimental trial was conducted in 2018 in Gioiosa Ionica (Reggio Calabria, Southern Italy, 38°21'12.59 "N; 16°20'42.03 "E; 195 m a.s.l.) on a sandy loam soil with a good content of phosphorus, potassium and organic matter, which main characteristics are reported in Table 1. Three different American hop varieties were studied: Cascade, Chinook and Comet (Table 2). The hop plants were purchased from the online company MRHOPS, which is specialised in potted hop seedlings. In March, the soil was milled burying 6.5 t ha^{-1} of mature manure. A 4 m high supporting structure was created, consisting of wooden poles with steel wires placed at the top, forming three rows. Irrigation was supplied when needed with a drip system. Seedlings were transplanted on

17 March, with a planting pattern of 3 m between rows and 1 m between plants, adopting a randomized block experimental design with 3 replications. Weeds were managed by hand. Ropes were arranged for each plant in a V-shaped structure to provide support for the growth of the climbing plants. Three to four shoots per plant were trained on each rope, while those in excess were not pruned. The plants did not undergo any pruning of excess shoots during the growing season. The ripe cones were harvested manually in a staggered manner between mid-July and mid-September, therefore dried in a desiccator at 50 °C, vacuum-packed and stored at 5°C. A One-way ANOVA model with multiple mean comparisons according to Tukey's (HSD) test was performed to determine differences between varieties, using DSAASTAT v. 1.1 software.[18] To ensure normality, percentage values were previously arcsin square root transformed; in tables, percentage data were reported. Meteorological data relating to the cultivation period were also acquired from an ARPACAL (Regional Agency for Environmental Protection of Calabria) data logger located near the experimental field. During the growing season, the temperature trend increased from February to July, at which the maximum temperature was recorded (37.2 °C) (Figure 1), temperature similar to the average of the maximum temperatures (37.6 °C) recorded in the two decades 1999-2018. The minimum temperature was instead recorded in March (6.1 °C). From March to August, rainfall was equal to 161 mm, basically lower than that recorded in the two decades 1999-2018, in the same period (184 mm). For what has been said above, frequent irrigation was used to ensure an optimal water supply to the hop plants, a plant notoriously demanding of water.

Field measurements

The following measurements were carried out on plants and inflorescences of each replication during the entire crop cycle: date of the main phenological phases (start of flowering, appearance of the first cones and cones ripening), plant height (cm), leaf length (cm) and width (cm), number of leaves per plant (on three plants for each replication), number of shoots per plant, length (cm) and width (cm) of cones, fresh and dry weight of the harvested cones (t ha^{-1}). Morphological measurements on leaves and cones were carried out on 10 leaves and cones for each replication. Cones were picked at maturity, when flowers presented a deep green colour, with a paper-like consistency to the touch, well-developed yellow lupulin glands and a strong and distinct aroma.

Essential oil extraction

Dry plant material of each sample (50 – 100 g) has been finely ground, placed in distilled water and submitted to hydrodistillation by a Clevenger apparatus as reported in the European Pharmacopoeia.[19] Three hydrodistillations lasting about three hours until there was no significant increase in the volume of oil collected have been carried out obtaining the following yields as % v/w: Cascade Gioiosa 0.80%, Comet Gioiosa 1.33%, and Chinook Gioiosa 1.24%. Each oil has been treated with sodium sulphate to eliminate any trace of water and stored at -20 °C until their analyses.

Essential oil analysis

Gas chromatographic (GC) analyses were run on a Shimadzu gas chromatograph, Model 17-A equipped with a flame ionization detector (FID), and with an operating software Class VP Chromatography Data System version 4.3 (Shimadzu). Analytical conditions: SPB-5 capillary column (15 m x 0.10 mm x 0.15 μm), helium as carrier gas (1mL/min). Injection in split mode (1:200), injected volume 1 μL (4% essential oil/CH_2Cl_2 v/v), injector and detector temperature 250 e 280 °C, respectively. Linear velocity in column 19 cm/sec. The oven temperature was held at 60 °C for 1 minute, then programmed as reported previously.[20] Percentages of compounds were determined from their peak areas in the GC-FID profiles.

Gas-chromatography-mass spectrometry (GC-MS) was carried out in the fast mode on a Shimadzu GC-MS mod. GCMS-QP5050A, with the same column and the same operative conditions used for analytical GC-FID, operating software GCMS solution version 1.02 (Shimadzu). Ionization voltage 70 eV, electron multiplier 900 V, ion source temperature 180 °C. Mass spectra data were acquired in the scan mode in *m/z* range 40-400. The same oil solutions (1 μL) were injected with the split mode (1:96).

Identification of Components of Essential Oils

The identity of components was based on their GC retention index (relative to C_9-C_{22} *n*-alkanes on the SPB-5 column), computer matching of spectral MS data with those from NIST MS libraries,[21] the comparison of the fragmentation patterns with those reported in the literature[22] and, whenever possible, co-injections with authentic samples.

RESULTS AND DISCUSSION

Agronomic traits

Crop cycle

Among the studied varieties, since the time of planting (17 March), Cascade showed the earliest vegetative growth with well-developed buds; Comet had a few hints of buds, while Chinook developed them exclusively at ground level. In the following period until mid-May, Cascade and Comet showed almost constant development. In contrast, Chinook plants exhibited uneven and slow growth. First inflorescences have been detected on Chinook variety (26 May) and in a later period on the remaining two varieties (01 July). Due to the different vegetative development and consequently to the different timing of strobiles differentiation, the harvest period resulted staggered among varieties starting from mid-July, with Chinook being the earliest, as stated above. The other varieties were harvested one month later Chinook. The varieties have shown times of development and maturation of the inflorescences contrasting with what is reported in the literature, probably due to the environmental conditions very different from the environments in which they are traditionally grown.

Morphological traits

Comet and Cascade showed the greatest and the lowest plant height development respectively, the former reaching an average height of 3.7 m and the latter 2.2 m (Figure 2). Chinook plants were in an intermediate position, reaching an average height of 3.0 m. Comet showed a fast growth rate, developing the highest plant biomass due to the numerous side shoots broadened by the plants.

On average, Cascade developed the higher number of stems per plant (8) measured at the end of the growth cycle, despite not being the most productive variety. Chinook and Comet, on the other hand, developed an average of 5 stems per plant.

Comet showed the highest number of leaves (78) per stem, followed by Cascade (62) and Chinook (62), following the height development of the plants.

The values of leaf length and width showed some variability. Chinook plants presented on average the highest values for both parameters, with a maximum value of 7.8 cm for length and 7.6 cm for width. Comet and Cascade plants, on the other hand, presented lower values and were quite similar. In the light of the results described here, it can be stated that these values are strongly related to the characteristics of the varieties.

At harvest, Chinook variety showed the longest average length (3.17 cm) and the widest average width (1.92 cm) of flower cones (Figure 3). The lowest values were found in the Cascade variety, with an average length of 2.37 cm and an average width of 1.42

cm. Comet developed cones with an average length of 3.14 cm and an average width of 1.50 cm.

These data are in agreement with those reported by Mongelli et al.[23], who, in a characterization of 23 wild genotypes of hop from Northern Italy, found a cone length ranging from 2.04 to 3.62 cm and a cone width ranging from 1.54 to 2.62 cm.

Considering the cone length/width ratio, the Comet variety presented the highest value (2.06), while the Cascade and Chinook varieties showed a very similar average value for this parameter of approximately 1.6. This ratio is an index of the shape of the flower cones, where a high value corresponds to tapered cones and a lower value to squat cones. Little information is reported in the literature, anyway, the three studied varieties showed a different combination of the cone and leaf dimensions allow us to hypothesize to use these data as useful tools to discriminate varieties.

Productive traits

Cone yield varied among varieties (Fig. 3). Comet proved to be the most productive variety, with a dry cone production of 0.35 t ha^{-1}, as the plants developed numerous lateral shoots on which numerous inflorescences were formed. The remaining varieties gave a similar yield of about 0.19 t ha^{-1}.

It should be noted that the productions obtained are relative to the first year of cultivation and that the plant increases its productivity in the years to come. Furthermore, in order to carry out the morphological surveys, the plant was left to grow freely without pruning the secondary stems, a circumstance that tends to reduce the vigour of the main ones.

The varieties were not very productive when compared with the typical yields of the same varieties where they are traditionally cultivated (1.5-2 t ha^{-1}).[7,16,24,25] This should not cause any perplexity, as these are productions relating to the first year of cultivation. Generally, full production is obtained in the third year for European cultivars, while good results are obtained in the first or second year for American ones.[26] Moreover, it should be noted that numerous variables can affect the yield. In fact, various studies and experiments conducted in Italy have shown how cultivars, cultivation techniques, soil, and climatic conditions can significantly influence production.[26] Furthermore, it should be remembered that deliberately, in order to detect the morphological characteristics, the plants did not undergo any pruning of the excess stems, which certainly contributed to reducing the vigour of the main stems. In fact, referring to the common management of a hops grove, it is a consolidated practice to choose two or three stems for each support wire, so as to promote an optimal growth density in order to obtain a high cone yield and a good quality.[1]

Experimental trials carried out in a Mediterranean environment on the agronomic and qualitative traits revealed that plant productivity is strongly influenced by the cultivar in a trial carried out on four varieties in the USA,[7,27] found yields ranging from 0.55 to 4.68 t ha^{-1}, in relation to different deficit irrigations. On the other hand Ceh (2014) 28 recorded yields from 0.95 to 1.51 t ha^{-1}. Anyway, our data are in agreement with those of some varieties reported by Rossini et al.[3] in a trial carried out in central Italy.

Essential oils

Table 3 lists the 92 compounds fully characterized in the three essential oils, whereas Figure 4 reports the GC profiles of the three oils. For an easier comparison of the oils the components have been grouped into five classes: monoterpene hydrocarbons, oxygenated monoterpenes, sesquiterpenes, diterpenes and others (not terpenoidic compounds). From a general point of view concerning the essential profiles obtained in this study the hop cultivated in Southern Italy is quite aligned with the hop samples cultivated in other Italian areas.[29,30]

In all samples monoterpene hydrocarbons is the main class ranging between 43 and 66 % *ca*., with only 9 components. Myrcene is largely the main compound in all cases, the other components are below 1% with the sole exception of β-*E*-ocimene which reaches 2% *ca*. in the Comet sample.[31,32] Very low is the number and quantity (8 and < 2%, respectively) of oxygenated monoterpenes.

Sesquiterpenes show a more complex and variegated composition: 31 compounds have been detected and their total amount ranges between 26 and 45%, being complementary to that observed for monoterpenes The cyclic sesquiterpenes β-caryophyllene and α-humulene are normally the main components of hop essential oils (together with the monoterpene hydrocarbon myrcene as previously mentioned). β-Caryophyllene is probably the most widespread sesquiterpene in Nature, α-humulene is present in many plants but not as widespread as the previous one, however, in the same essential oil hop samples it can reach almost half of the total oil.[29,33] In the case here examined β-caryophyllene is one of the main components in all samples (range 5 – 10 %

ca.); much higher is the concentration of α-humulene (range 12 – 20% *ca.*), placing itself in second place for quantity among all components, however, in the Comet sample this compound does not reach 1%, therefore, then it can be counted a low humulene variety.[33] Always among the sesquiterpenes appreciable amounts are showed by β- ed δ-selinene, which in Comet reach about 5%, whereas in the other two samples their content ranges between 1 and 2%, respectively. Another interesting group of sesquiterpenes is represented by γ- δ- and α-cadinene, which in the Chinook sample reaches appreciable amounts (Table 3), whereas in the other two samples remain below 1%.

Finally, the class denominated 'others', namely, the not terpenoidic compounds, ranges between 5 and 8 % of the total amount, despite being the most numerous with 42 components, most of them below 1%.

Given the important and essential role of hop in beer production, either for the bitter substances and for the aromatic compounds, several researches have been carried out to understand the different aromatic profiles of the hop varieties, but also to valorize a particular territory and/or production area.[32,34] The data until now collected, included those coming from the present study, do not allow to establish yet if Southern Italy may be considered a particularly favourable area, however, some aspects emerged also from this study leave hope for the future.

CONCLUSION

The results obtained from the experimental trial conducted on the three varieties of American hops, *Humulus lupulus* L., allowed us to draw some conclusive considerations regarding their morphological, phenological and chemical characteristics displayed in cultivation in the Calabrian environment. First of all, it is possible to state how hop introduced into cultivation for the first time in the Calabrian coastal area showed excellent adaptability to the tested environment.

As for the studied characteristics, a certain variability was found regarding the morphological traits and the yield in flowering cones. Cone and leaf dimensions seem to be interesting criteria to discriminate varieties. The pedoclimatic conditions of the chosen area have certainly influenced the development of the plants, especially if we compare the typical yields generally obtained in the countries of traditional cultivation of the crop with those obtained in this experimental test, the latter being basically lower. In particular, Comet was the most productive variety, while Chinook emerged as the earliest variety, followed by Comet and Cascade.

The promising results obtained in this first experience on the introduction into cultivation of hop in Southern Italy, laying the foundation for a widespread of cultivation, and develop the craft industry with high traceability and zero km hop.

CONFLICT OF INTEREST

There are no conflicts of interest to declare.

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Table 1. Soil main physical and chemical traits of the experimental field.

Trait	value
Gravel >2 mm (%)	26.4
Sand (2–0.05 mm) (%)	64.5
Silt (0.05–0.002 mm) (%)	22.7
Clay (<0.002 mm) (%)	12.7
ECe (dS m^{-1})	0.153
pH	6.8
Total CaCO3 (g kg^{-1})	0
Active CaCO3 (g kg^{-1})	0
Organic matter (g kg^{-1})	22.5
Total N (g kg^{-1})	0.8
C/N	16.3
Available P as P_2O_5 (ppm)	98
Exchangeable K as K_2O (ppm)	248
Exchangeable Ca as CaO (ppm)	2515
Exchangeable Mg as MgO (ppm)	288
Exchangeable Na (ppm)	5
Mg/K (meq/100 g)	2.71
CEC (meq/100 g)	17.8
BS (%)	62
ESP (%)	0.12

Table 2. Maturation, use and provenance of the studied varieties.

Variety	Maturation	Use	Origin
Cascade	Medium	Bittering/aroma	USA
Chinook	Medium-late	Bittering/aroma	USA
Comet	Medium-late	Bittering/aroma	USA

Table 3. Chemical Composition of the Three Hop Essential Oils.

#a	RI[b]	RI[c]	Class/Compound	CASCADE		CHINOOK		COMET	
				%	SD	%	SD	%	SD
			Monoterpene hydrocarbons (#9)	64.16	1.32	43.42	2.32	66.13	2.11
6	934	930	α-Pinene	0.18	0.02	0.15	0.00	0.24	0.01
8	957	954	Camphene	0.04	0.00	-	0.04	0.00	
10	976	979	β-Pinene	1.03	0.06	0.62	0.01	0.93	0.02
11	**993**	**991**	**β-Myrcene**	**62.04**	**1.33**	**41.89**	**2.29**	**61.76**	**2.25**
12	1001	1003	α-Phellandrene	0.04	0.00	0.06	0.00	0.07	0.00
16	1029	1029	Limonene	0.59	0.01	0.56	0.04	0.80	0.02
17	1037	1035	β-Z-Ocimene	0.03	0.00	0.03	0.00	0.10	0.02
18	1048	1050	β-E-Ocimene	0.17	0.04	0.07	0.01	2.14	0.27
20	1059	1060	γ-Terpinene	0.04	0.02	0.03	0.00	0.05	0.02
			Oxygenated monoterpenes (#10)	**1.87**	**0.08**	**1.59**	**0.04**	**1.79**	**0.09**
23	1098	1097	Linalool	0.37	0.01	0.34	0.01	0.56	0.02
25	1115	1119	1-Myrcenol	0.11	0.01	0.16	0.01	0.02	0.00
28	1184	1177	Terpinen-4-ol	0.04	0.01	0.02	0.00	-	
33	1240	1238	Neral	-	0.04	0.01	t		
34	1275	1267	Geranial	0.06	0.00	0.05	0.00	0.04	0.00
39	1327	1324	Methyl geranoate	0.42	0.03	0.56	0.01	0.45	0.05
44	1384	1381	Geranyl acetate	0.32	0.02	0.04	0.01	0.21	0.01
51	1474	1477	Geranyl propionate	0.16	0.02	0.05	0.01	0.34	0.02
60	1519	1515	Geranyl isobutanoate	0.39	0.04	0.33	0.02	0.17	0.01
			Sesquiterpene hydrocarbons (#21)	**26.61**	**0.13**	**41.50**	**0.22**	**23.72**	**0.15**
40	1345	1351	α-Cubebene	0.03	0.00	0.02	0.00	-	
42	1377	1375	α-Ylangene	0.09	0.00	0.19	0.01	0.05	0.00
43	1381	1377	α-Copaene	0.13	0.01	0.67	0.03	0.06	0.01
45	**1426**	**1419**	**β-Caryophyllene**	**5.28**	**0.26**	**7.97**	**0.50**	**9.47**	**0.68**
46	1430	1432	β-Cubebene	t	0.31	0.03	t		
47	1434	1434	α-*E*-Bergamotene	0.12	0.01	0.04	0.00	0.05	0.00
49	1440	1456	β-*E*-Farnesene	0.15	0.00	0.02	0.00	0.03	0.01
50	**1462**	**1455**	**α-Humulene**	**12.12**	**0.85**	**19.41**	**1.44**	**0.48**	**0.04**
52	1478	1476	*E*-Cadina-1(6),4-diene	t	0.19	0.01	t		
53	1481	1480	γ-Muurolene	0.72	0.04	1.82	0.29	0.97	0.08
54	1485	1484	α-Amorphene	0.06	0.00	0.15	0.01	0.05	0.00
55	1492	1490	β-Selinene	1.78	0.10	1.43	0.04	4.79	0.39
56	1494	1495	Cadina-1,4-diene	0.25	0.07	t	t		
57	1500	1492	δ-Selinene	2.16	0.16	2.13	0.37	5.08	0.42
58	1507	1500	α-Muurolene	0.12	0.03	0.36	0.16	0.37	0.04
59	1512	1506	β-Bisabolene	0.26	0.06	0.21	0.05	0.41	0.02
61	1524	1513	γ-Cadinene	0.05	0.01	1.65	0.10	0.15	0.01
62	1526	1522	7-epi-α-Selinene	-	0.07	0.00	-		
63	1528	1523	δ-Cadinene	0.81	0.08	3.34	0.19	0.37	0.03
64	1542	1538	α-Cadinene	0.32	0.05	1.64	0.01	1.27	0.09
65	1549	1546	Selina-3,7-(11)-diene	0.27	0.04	1.39	0.01	1.15	0.10
			Oxygenated sesquiterpenes (9)	**3.31**	**0.05**	**2.93**	**0.04**	**1.99**	**0.02**
66	1578	1572	Caryolan-8-ol	0.13	0.01	0.11	0.00	0.13	0.01
67	1589	1583	Caryophyllene oxide	0.67	0.06	0.21	0.08	0.14	0.01
68	1616	1608	Humulene epoxide	1.01	0.18	0.48	0.15	t	
69	1621	1619	1,10-di-epi-Cubenol	0.08	0.01	0.10	0.01	0.15	0.01

70	1635	1628	1-epi-Cubenol	0.05	0.00	0.13	0.01	0.05	0.00
71	1638	-	N.I.S.d	0.07	0.01	0.16	0.01	0.11	0.01
72	1648	1640	epi-α-Cadinol	0.19	0.03	0.48	0.04	0.13	0.01
73	1658	1646	α-Muurolol	t	0.05	0.00	0.10	0.01	
74	1663	-	N.I.S.e	1.11	0.16	1.21	0.03	1.18	0.08
			Diterpenes (#2)	**0.22**	**0.07**	**0.10**	**0.05**	**0.03**	**0.00**
75	1950	1955	*m*-Camphorene	0.03	0.00	0.05	0.02	-	
77	1974	1986	*p*-Camphorene	0.19	0.06	0.05	0.01	0.03	0.00
			Others (#27)	**3.33**	**0.41**	**6.53**	**0.33**	**2.85**	**0.17**
1	855	855	2-*E*-Hexenal	0.04	0.00	-	-		
2	867	875	2-Methylbutyl acetate	-	0.02	0.00	0.05	0.01	
3	899	905	Heptanal	0.03	0.00	0.02	0.00	0.03	0.00
4	911	911	Isobutyl isobutyrate	0.03	0.00	0.09	0.01	0.04	0.00
5	923	927	Methyl hexanoate	0.03	0.00	0.06	0.01	0.03	0.00
7	939	939	Allyl isovalerate	t	-	0.03	0.00		
9	970	966	Isoamyl propionate	0.13	0.02	0.49	0.07	0.17	0.01
13	1010	1009	Isoamyl isobutyrate	0.07	0.00	0.32	0.02	0.06	0.01
14	1014	1015	2-Methylbutyl isobutyrate	0.16	0.01	0.92	0.07	0.20	0.01
15	1023	1025	Methyl heptanoate	0.19	0.01	0.14	0.01	0.17	0.01
19	1054	1063	2,3,6-trimethyl-1,5-Heptadiene	0.04	0.00	0.02	0.00	0.02	0.00
21	1086	1087	methyl 6-Methylheptanoate	0.13	0.02	0.41	0.08	0.25	0.02
22	1092	1090	2-Nonanone	0.19	0.02	0.03	0.01	0.12	0.00
24	1101	1100	Nonanal	0.18	0.03	0.17	0.01	0.09	0.03
26	1124	1127	Methyl octanoate	0.11	0.01	0.16	0.02	0.10	0.01
27	1179	1192	2-Decanone	0.05	0.00	0.03	0.00	0.08	0.03
29	1192	1229	Methyl nonanoatef	0.06	0.01	0.04	0.01	0.27	0.01
30	1195	1192	Dodecane	0.03	0.01	0.31	0.05	t	
31	1204	1201	Decanal	0.03	0.00	0.02	0.00	-	
32	1225	1223	Methyl 4-nonenoate	0.09	0.01	0.13	0.00	0.11	0.00
35	1280	1271	2-*E*-Decen-1-ol	0.06	0.01	0.05	0.00	0.11	0.00
36	1312	1289	Methyl 4-decenoatef	0.42	0.08	0.92	0.12	0.62	0.06
37	1290	1294	2-Undecanone	0.04	0.01	-	0.08	0.01	
38	1313	1323	Methyl caprate	0.03	0.01	0.20	0.05	0.03	0.00
41	1367	1383	2-Dodecanone	0.04	0.00	0.06	0.02	0.02	0.00
48	1440	1410	3-*Z*-Decen-1-ol acetatef	0.15	0.03	t	0.06	0.01	
76	1952	1959	Hexadecanoic acid	0.21	0.04	0.08	0.01	-	
78	2650	2682	Lupulon	0.20	0.12	1.03	0.22	0.11	0.01
			α-Humulene/β-Caryophyllene	***2.29***	***0.09***	***2.43***	***0.03***	***0.05***	***0.00***

[a]The numbering refers to elution order, and values (relative peak area percent) represent averages of 9 determinations (t = trace, < 0.05%), [b] Retention index (RI) relative to standard mixture of n-alkanes on SPB-5 column; [c] Literature Retention Index (RI); [d] N.I.S. = Not Identified Sesquiterpene MW: 222 $C_{15}H_{26}O$ (m/z = 43, 67, 79, 93, 109, 135, 164, 204); [e] N.I.S. = Not Identified Sesquiterpene MW: 222 $C_{15}H_{26}O$ (m/z = 43, 59, 81, 105, 119, 133, 161, 179, 204); [f] tentatively identified.

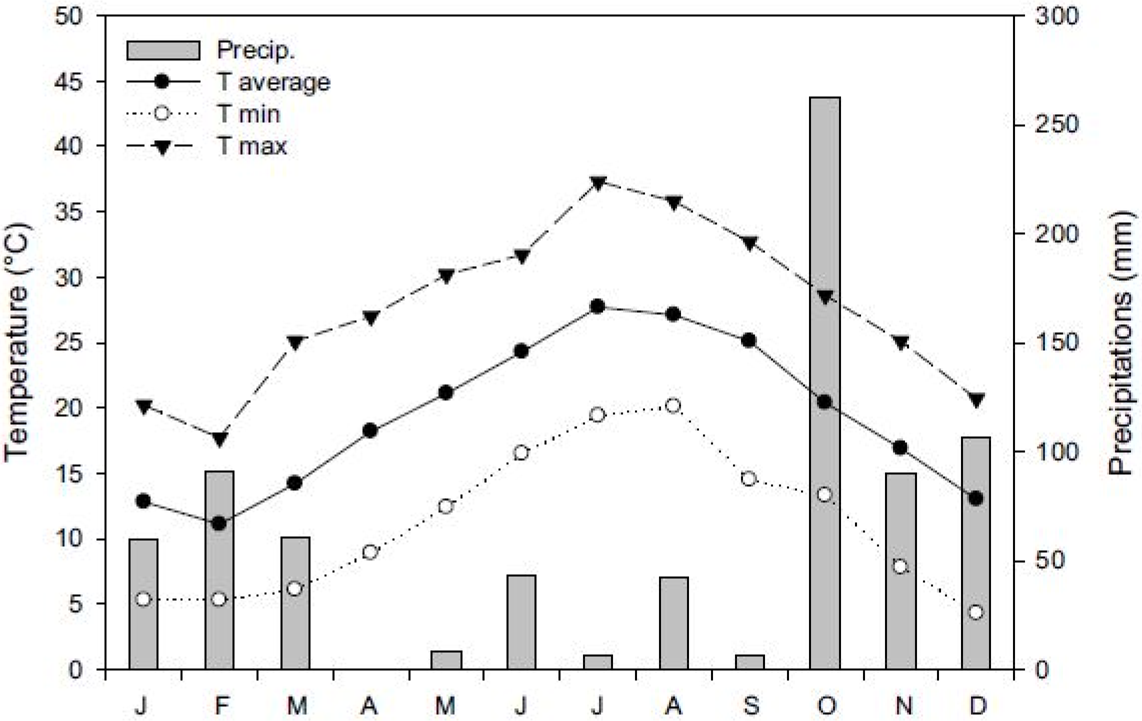
Figure captions

Figure 1. Meteorological trend of 2018 at the experimental station of Gioiosa Jonica (RC).

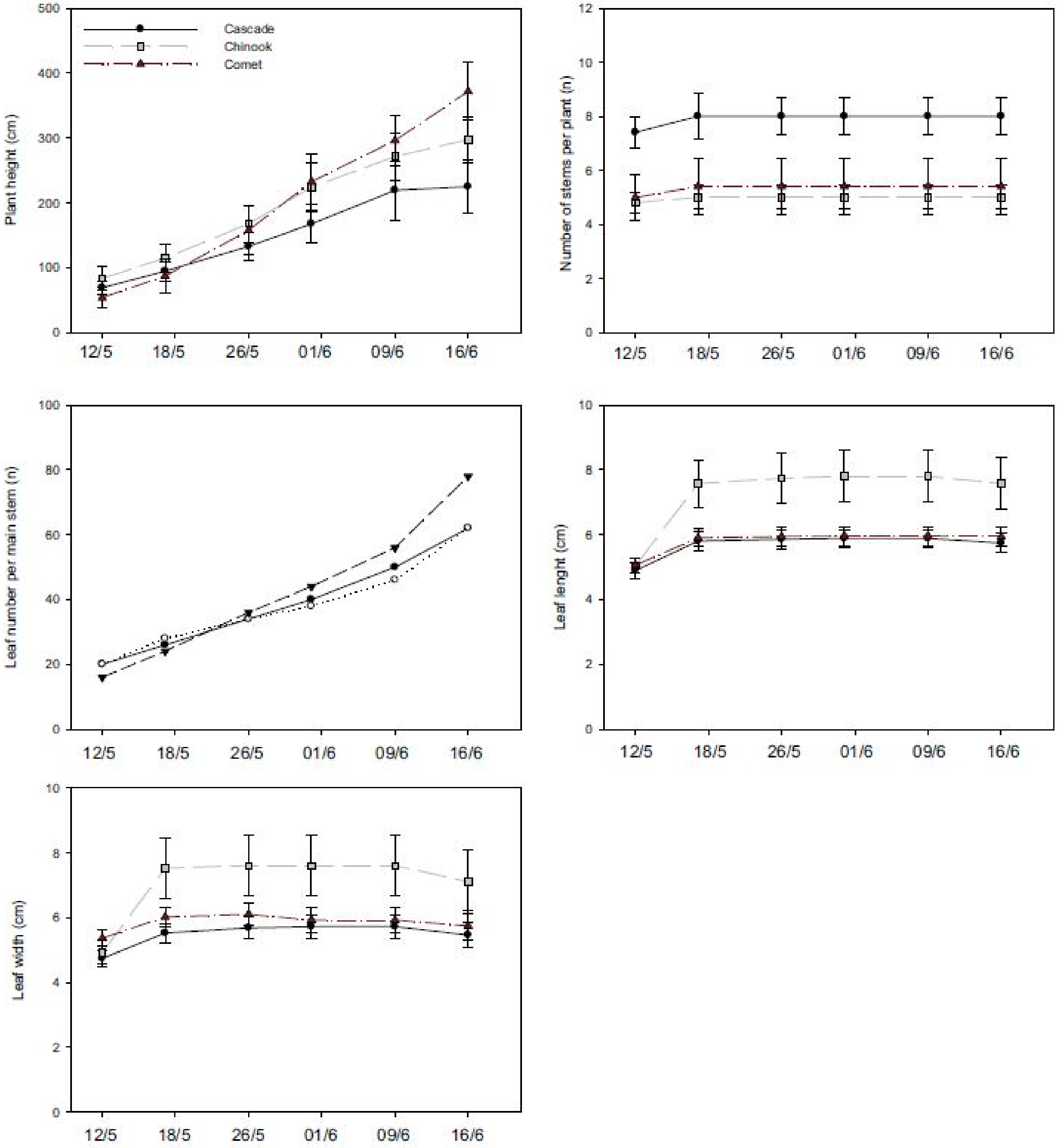
Figure 2. Plant height, number of stems per plant, number of leaves per plant, leaf length and leaf width (± Standard Error) of the three studied varieties during the crop cycle.

Figure 3. Flower cones length (Cm), width (Cm), length/width ratio and yield (T ha^{-1}) of the studied varieties. Different letters indicate significative differences ($P < 0.01$) between varieties.

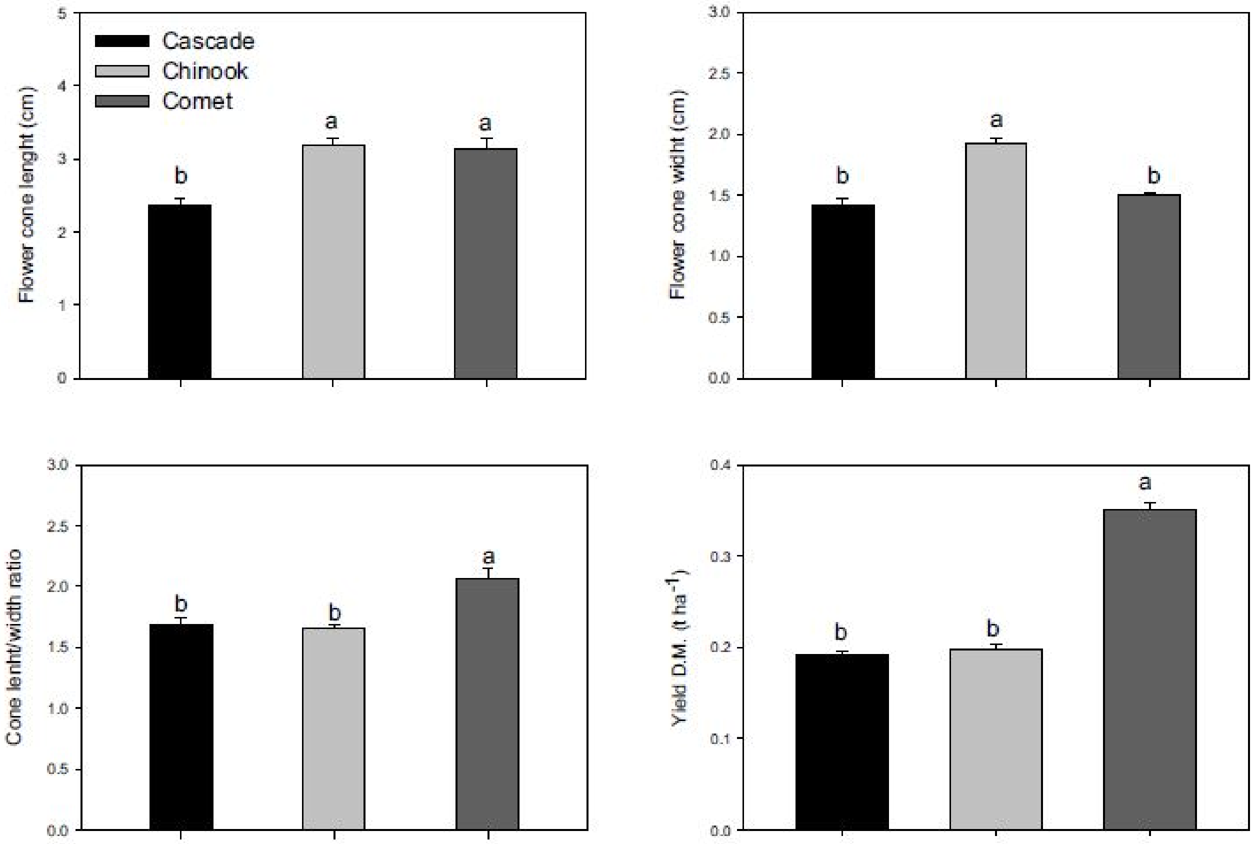
Figure 4. GC profiles of Cascade, Chinook and Comet Hop essential oils (For numbering see Table 3).



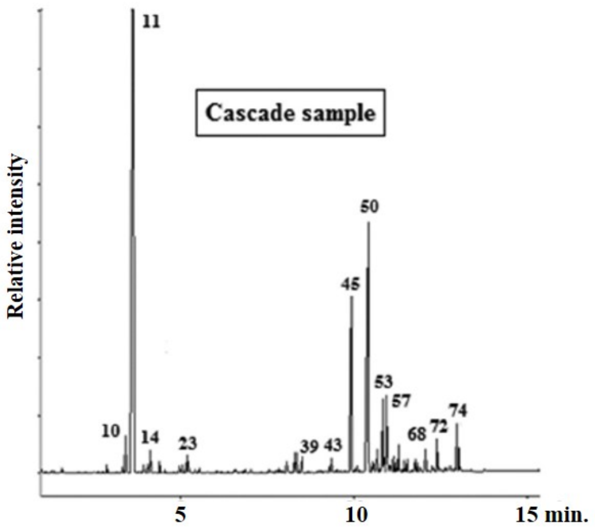
Precip.
T average
T min
T max
Temperature (°C)
Precipitations (mm)
50
45
40
35
30
25
20
15
10
5
0
300
250
200
150
100
50
0
J
F
M
A
M
J
J
A
S
O
N
D

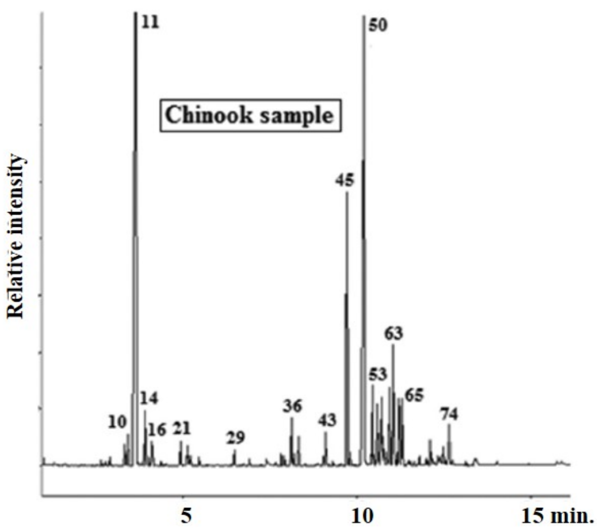


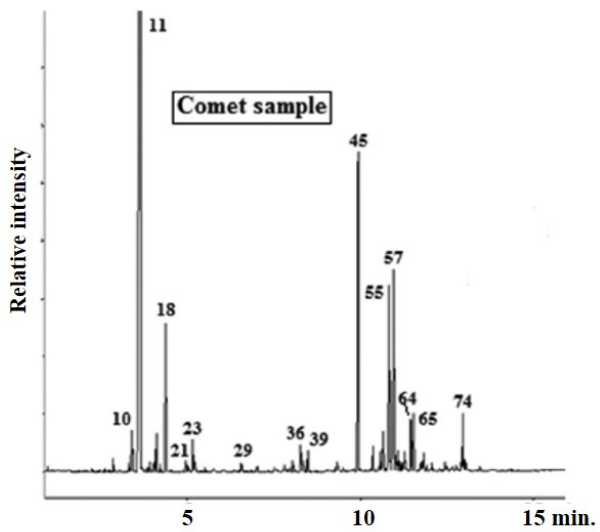
Cascade
Chinook
Comet
Plant height (cm)
Number of stems per plant (n)
Leaf number per main stem (n)
Leaf lenght (cm)
Leaf width (cm)
12/5
18/5
26/5
01/6
09/6
16/6



Cascade
Chinook
Comet
Flower cone lenght (cm)
Flower cone widht (cm)
Cone lenht/width ratio
Yield D.M. (t ha-1)
0
1
2
3
4
5
0.0
0.5
1.0
1.5
2.0
2.5
3.0
0.1
0.2
0.3
0.4
a
b


Cascade sample
Relative intensity
11
50
45
53
57
10
14
23
39
43
68
72
74
5
10
15 min.


Chinook sample
Relative intensity
11
50
45
63
53
65
14
10
16
21
29
36
43
74
5
10
15 min.


Comet sample
Relative intensity
11
45
57
55
18
64
65
74
10
23
21
29
36
39
5
10
15 min.