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*Research Article*

# Oleoresin tapping of *Pinus pinaster* Ait. in the Cévennes, a region of France: Impact of location on oleoresin production and composition

**Peggy Rigou[1], Ambre Bouchard[2], Laura Sigler[1], Virginie Guiroy[3], Margot Lepetit[4] and Pascale Chalier[2]***

1. SPO, University of Montpellier, INRAE, Institut Agro, 34060, Montpellier, France.
2. IATE, University of Montpellier, INRAE, Institut Agro, 34060 Montpellier, France.
3. Syndicat mixte du Pays des Cévennes, Charte Forestière, 30100 Alès, France.
4. Charte Forestière de Territoire Sud Lozère, 48400 Florac Trois Rivières, France.

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***Corresponding Author***
Prof. Dr. Pascale Chalier
E-mail:
pascale.chalier@umontpellier.fr
Tel.: +33683424459

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## Abstract

The aims of this study were to compare the resin yields of *Pinus pinaster* Aiton and byproduct composition depending on location. Chareneuve and Vern, both sites located in the Cévennes, a region of southeastern France, were gemmed. Chareneuve site, with larger trees and lower tree density, showed a higher oleoresin yield (approximately 4 kg /tree) compared to Vern (approximately 2.79 kg/tree). For both sites, the highest oleoresin yields were recorded when the temperature during the day exceeded 28°C. Although turpentine essential oil yield was similar (~30%) between the two sites, the EO composition varied: Vern EO had more β-pinene (19%) against 13 % for Chareneuve, resulting in a lower α-/β-pinene ratio (4) compared to Chareneuve (6.7). Next to pinenes, four other compounds: δ-3-carene, β-phellandrene, longifolene and β-caryophyllene, allowed differentiation between the two sites. In contrast, the rosin composition showed minor differences between the two locations. Abietane-type acids were found to be more abundant than pimarane-types, consistent with Mediterranean climates. The oleoresin yields and chemical differences in the composition of EO between these two targeted sites, highlight the need for further studies across more sites but also tapping years.

## 1. Introduction

Oleoresin synthesis in many conifer species, including *Pinus pinaster Ait*, serves as a defense mechanism, which can be either constitutive or induced by external factors like pathogen and insect attacks [1].This substance is produced in specialized secretory cells during tissue development and accumulates in all parts of the tree such as stems, roots, branches, needles and cones [2]. Resin production can be simulated through various techniques known as tapping, which involves notching the tree, sometimes with an activating agent [3-5]. The quantity of resin produced is influenced by the tapping method, chemical inducers, climate variation, and soil composition [6-9]. Oleoresin is composed of two main parts; a volatile part (25 to 30%) known as the turpentine essential oil, characterized by volatile monoterpenes (especially pinene isomers) and some sesquiterpenes, and a solid part (70-75%) called rosin, which is primarily composed of diterpenic acids (90%), such as abietane and pimarane acids [10, 11].

Oleoresin by-products have been used since ancient time for a broad range of applications, such as boat caulking and in the production of soap, printing ink, oil paint, fuel, synthetic rubber, adhesives, pesticides, aroma, emulsifiers, perfumes and medicines [7, 12-14]. Oleoresin and its sub-products are considered as sustainable forest products and their diverse applications have increased interest from industries and consumers potentially leading to a revival in their production. All countries are confronted with the need to revitalize forests from both an economic and environmental perspectives [9, 15]. Notably, pine forests are described as efficient for carbon sequestration, particularly when tapping is performed [16] In France, *Pinus pinaster* Ait is the dominant species, growing on 1,050,000 hectares, with 740,000 hectares being pure stand (2017 - IGN forest inventory, https://inventaire-forestier.ign.fr/essence/pin_maritime/). While tapping was a well-established activity in France at the beginning of the last century, with a resin production reaching 180,000 tonnes [17], it declined and disappeared by 1990 due to competition from other countries such as Brazil and China.

Currently, tapping is recommencing in France, employing new methods that are more respectful of the trees, such as the eco-certified paste developed by Biogemme®, particularly in the oceanic southwest region known as "the Landes of Gascogne" which is the main source of *Pinus pinaster* oleoresin. However, the abundance of this species in other French forests, such as the Cévennes in the southeast, presents an opportunity to valorise this deindustrialized area. Indeed, *Pinus pinaster* was extensively introduced to the Cévennes in the mid-19$^{th}$ century by mining companies, for industrial purposes, i.e. shoring up coal mine galleries. This choice was due to the rapid growth of this pioneer species, its adaptation to the local soil and full acclimatization. With the end of mining, there is an urgent need to enhance the value of this species in this area. Indeed, the quality of wood is generally poor, and it is mainly used for energy, paper pulp production and pallet sawing purposes, which represents little added value or, the best qualities being keept (timber). given the renewed interest in natural products from the forest, the creation of a value chain for the Cévennes maritime pine resin though tapping has been envisioned and supported by local organisations (Charte Forestière de Territoire Sud Lozère Lozère, Charte forestière, Syndicat des pays mixtes de Cévennes). This initiative necessitates knowledge about the trees' capacity for resin production and the variability in yield and composition. There are significant differences in the oleoresin composition among various species (e.g. *Pinus Elliotti, Pinus nigra,* and *Pinus halepensis*) among the number of components (qualitative composition) within the same tree species remains relatively stable [7, 8, 18-21]. However, the quantitative composition can be influenced by genetic differences among populations, and variations between individuals in the same population or under developmental and environmental stimuli [22, 23]. For *Pinus pinaster* Ait**.**, various chemotypes have been distinguished based on terpenic compounds serving as genetic markers dependent on location. Three distinct groups have been established [24]: the Atlantic group found in the Iberian Peninsula (Portugal and Galicia) and in the southwest of France (Landes of Gascogne), the Mediterranean group in south of Spain, southeast France and Corsica, Sardinia and Maghrebian (Morocco) [25, 26]. Although the pine seeds planted in the Cévennes originated from the Landes, the trees growing in the Cévennes have never been characterised in terms of chemotype. It is known that trees and their defence mechanisms can be modified by natural, climatic and anthropogenic events, as always observed for *Pinus pinaster* in the Iberian Peninsula [9, 25]. In another study, it was demonstrated that both the growth and resin yield of *Pinus pinaster* trees from Portugal increased with temperature but there was a trade-off between these two parameters [27]. The soil composition and climate in the Landes and Cévennes regions differ largely in terms of composition, precipitation and extreme temperature range [28, 29]. An initial study on resin harvested in the Cévennes showed similarities in EO composition with those obtained from the southwest [30], but the tapping time was short and potentially unrepresentative. Moreover, resin production can also be influenced by intrinsic factors, such as dendrometric characteristics: tree size or tree height (9), with larger trees generally producing more resin. Another important factor is silvicultural practices


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which affect trees growth [31]. The complexity of the factors influencing resin production and the research on economic viability need to apply model managements [31].

The objectives of the current study are as follows: (i) to compare the oleoresin production of *Pinus pinaster* growing in Cévennes from 2 different locations ii) to identify the major terpenes of the turpentine essential oil and the ratio between the two pinenes iii) to identify the main resin acids in rosin after distillation (iii) to investigate any correlations among the yield, the essential oil and rosin profile, the characteristics of the site and the trees diameter and (iv) to compare these findings with other sites production in southern Europe.

## 2. Materials and methods

### *2.1. The location characteristics*

The two sites differed in numerous parameters.

The Chareneuve site is a pure *Pinus pinaster* forest, located at the bottom of a valley at an altitude of 300 m with southeast-facing exposure. The tree density is estimated at 800 stems per hectare and the average tree height is around 18 meters. The soil was composed of gneiss.

The Vern station is a coniferous forest composed of a mix of *Pinus pinaster* and *Pinus nigra laricio* growing in a cedar plantation. The site is situated at the top of a valley at an altitude of 500 m, with a southern exposure. Tree density is estimated at 1,600 stems per hectare and the average tree height is around 16 meters. The soil consists of conglomerates composed of shale, sandstone and coal.

### *2.2. Oleoresin harvesting and samples*

Oleoresin samples were collected following the tapping of *Pinus pinaster* trees in two privately owned forest stations located in the Cévennes region of France, specifically in the areas known as Chareneuve and Vern, within the commune of Chambon. 50 trees per station were gemmed using the Biogemme method developed and patented by Holiste® [11]. Initially, two circular incisions were made in the bark of each tree using a hole drill. A 100% natural organic acid activator (Holiste®) was applied to the breach, allowing the resin to flow abundantly and slowing down the tree's healing process. A plastic bag with a connector is maintained on the breath collecting the resin as it drains from the tree allowing the resin to be protected from impurities, evaporation and oxidation. The harvesting campaign commenced on May 6, 2022. Every three weeks, two new holes were drilled, the activator paste was reapplied, and the containers were reattached after weighing. If the containers were too full, new containers were placed. This operation was performed 6 times. At the end of the harvested (September 23rd, i.e. after 111 days or around 18 weeks), the total pine resin removed from each station and stored in a metal barrel until analysis.

A sample of oleoresin collected in the Landes was furnished by Holiste (Seignosse, France) without indication of a specific location. A sample of rosin furnished by Holiste and obtained after oleoresin distillation by Biolandes (Le Sen France) was also analysed.

### *2.3. Distillation of resin*

The essential oil content of the resin was rapidly estimated by weighing after heating in an oven at 150 °C for 30 min as described by Rubini et al. [8].

#### *2.3.1. Conventional Hydro-distillation (CHD)*

Turpentine essential oil was separated from rosin using a laboratory Clevenger apparatus. 50 g of resin and 100 mL of water (ratio water/resin equal to 2mL.g$^{-1}$) were added to the balloon and the hydro-distillation was carried out at 115 °C using a balloon, heated for 120 min. The turpentine essential oil was recovered in a test tube, kept in an ice bath, temporarily separated from residual water by density difference, and then dried on ammonium sulphate to remove residual water. The recovered turpentine essential oil was weighed and the yield expressed in % (w/w) as the ratio between the EO weight and the initial resin weight was calculated. The EOs were stored at 4 °C until analysis. Each assay was repeated three times.

#### *2.3.2. Vacuum Distillation (VD)*

VD was performed using a rotavapor® apparatus (R300, Büchi UK, LTD, United Kingdom) equipped with a vacuum pump, a recirculating chiller, a heater bath and regulated by a central interface. Around 50 g of homogenized resins were distilled in a 500 mL balloon rotated at 150 rpm for 60 min at 95 °C under a pressure of 1 KPa. The turpentine oil recovered in the solvent balloon was weighed to calculate the EO


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**Table 1.** Compounds specificities given by furnisher[a].

| Compounds name and CAS number | Molar mass ($g.mol^{-1}$) | Purity[a] (%) | Density[a] $kg.m^{-3}$ (25°C) |
|---|---|---|---|
| α-pinene 80-56-8 | 136 | 99 | 859 |
| β-pinene 127-91-3 | 136 | 99 | 860 |
| Camphene 79-92-5 | 136 | 96 | 842 |
| δ-3-Carene 13466-78-9 | 136 | 90 | 856 |
| D-limonene 123-35-3 | 136 | 95 | 842 |
| β-myrcene 5989-27-5 | 136 | 95 | 794 |
| Trans-β-caryophyllene 87-44-5 | 204 | 90 | 907 |
| Longifolene 1139-30-6 | 204 | 100 | 930 |

extraction yield, as mentioned above. The EOs were stored at 4°C until analysis. Each distillation was repeated three times.

*2.4. Analyses of essential oils*

*2.4.1. Chemicals and standard solution for calibration*

Hexane (99% purity), the dilution solvent of essential oil, 4-nonanol (99% purity and density 0.82), the internal standard and the different chemicals used for identification and calibration curves were purchased from Merck-Sigma-Aldrich (France). The names, purities, and mass densities of these compounds are listed in Table 1.

Standard stock solutions of $\alpha$ and β-pinene, D-limonene, β-myrcene, 3-δ−carene, camphene, trans-β-caryophyllene and longifolene were prepared in hexane. Standard solutions from 4 $mg{\cdot}L^{-1}$ to 85 $mg{\cdot}L^{-1}$ were prepared from the stock solutions in order to establish GC-MS calibration curves. 100 µL of 4-nonanol at 102 mg/L was added as an internal standard. The calibration curves were established by reporting the area ratio between the targeted aroma compound and the internal standard. All concentrations are expressed in $mgL^{-1}$.

*2.4.2. Analysis of turpentine EO by GC-MS*

Analysis and quantification of turpentine EO were carried out using a Gas Chromatography (GC-2010 plus) coupled to a Mass Spectrometer (QP2020) Shimadzu instrument (Shimadzu Corporation, Marnela Vallée, France). Several dilutions of EOs samples (100, 500, 1000) were analysed in order to fit into the linear response of the compounds, and to allow the quantification of 3-δ-carene, longifolene, D-limonene, β-myrcene, camphene, (E)-β-caryophyllene and $\alpha$ and β-pinene respectively. An SH-Rtx-Wax (Shimadzu) polar column (30 m, 0.25 mm, 0.25 µm thickness) was used for compound separation using helium as carrier gas at a flow rate of 1.2 mL/min. The injector temperature was set at 250 °C and injection was performed with a split ratio of 20. The GC oven temperature was programmed from 40 °C to 100 °C at a rate of 2 °C/min, then to 230 °C at a rate of 15 °C $min^{-1}$ and kept at 230 °C for 10 min. The transfer line and ion source temperatures were set to 230 °C. The MS was equipped with a simple quadrupole detector and used in electron impact mode with an ionization energy of 70 eV. Analyses were run in the full scan mode with a scan range between 40-500 amu. The identification of components was done by comparing with mass spectra libraries (NIST 17/Wiley) and confirmed with the standard solution. Linear retention indices were calculated using alkane standard solution and compared to data on Nist Webbook https://webbook.nist.gov/chemistry. Concentrations of the compounds were calculated using


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the corresponding calibration curves. Secondary compounds for which a calibration curve was not established were semi-quantified by comparing their peak area to the total area of the identified peaks.

*2.4.3. Density and refractive index measurement of essential oil*

The density was measured with a portable densimeter (Densito, Mettler Toledo) at 20 °C. The density of the EOs, expressed in $g.cm^{-3}$, must be between 0.860 and 0.872, as specified by the standard ISO-11020-1998 [32]. The measure precision was 0.001 $g.cm^{-3}$ and the resolution 0.0001 $g.cm^{-3}$.

The refractive index was measured with a portable refractometer (30GS, Mettler Toledo) at 20 °C. According to standard ISO-11020-1998 [32], the refractive index measured with the D-line of sodium, must be between 1.4650 and 1.4750. The precision of measurement was 0.0005.

*2.5. Rosin analyses*

*2.5.1. Chemicals, methylation and calibration curves*

Abietic acid (CAS 475-20-7, purity 75%), isopimaric acid (CAS 5835-26-7, purity >98%) and dehydroabietic acid (CAS 1740-19, purity > 90%) were purchased from Merck, France.

A standard stock solution of abietic acid was prepared by dissolving 3120 mg in 10 mL of ethyl alcohol. Standard calibration of abietic acid was carried out in the range of 624-1560 $mgL^{-1}$ by adding the appropriate volume (20, 30, 40, 50 µL) of a stock solution (31.2 $gL^{-1}$). A 100 µL of tetra methyl hydroxyl ammonium (TMHA) as derivatization agent and 100 µL of 4-nonanol (102 $mgL^{-1}$) solutions were used as internal standard, and the final volume was adjusted to 1 mL using ethyl alcohol. The purity of abietic acid (75%) was considered to establish the calibration curve, expressed as the ratio of IS and compound area as a function of abietic acid concentration. The same calibration was used to quantify the different diterpenic acids. Isopimaric and dehydroabietic acids solution was prepared to confirm the identification of these components in the samples.

The same protocol of derivatization was used for the samples: 0.4 g of rosin was solubilized in 10 mL of ethyl alcohol and, then 20 and 50 µL of this solution was methylated by adding 100 µL of TMAH and 100 µL of 4-nonanol solutions The final volume was adjusted to 1 mL using ethyl alcohol.

*2.5.2. Analysis and quantification of rosin by GC-MS*

Analysis and quantification of rosin were carried out using the same column and GC-MS equipment used for EO. The only change is the GC and MS method, the helium flow was fixed to 1 mL $min^{-1}$, the oven temperature was programmed from 40 °C to 200 °C at a rate of 15 °C/min, then to 230 °C at a rate of 3 °C $min^{-1}$ and kept at 230 °C for 15 min. The injector temperature was set at 250 °C and injection was performed with a split ratio of 20. The transfer line temperature was set to 230 °C and the ion source temperature was 250 °C.

The MS was used in the electron impact mode with 70 eV of ionization energy. Analysis ran in the full scan mode with a scan range between 40-500 amu. The identification of components was performed by comparing with the mass spectra of libraries (NIST 17/Wiley) and literature. The percentage of each component was calculated from their specific area reported to the total area and quantified using a calibration curve.

*2.5.3. Acid index*

The acid number was determined by dissolving the rosin (0.300 g) in neutralised ethyl alcohol (5 mL) and then titrating the acids present with a 0.1 N ethanolic potassium hydroxide solution in presence of phenolphthalein (ASTM D1240-14) [33]. The acid value was expressed in mg of KOH per gram of rosin.

*2.6. Statistical analysis*

One-way ANOVA tests with one factor were used to assess the significance of the differences. When significant differences were revealed ($p < 0.05$), mean values were compared using Tukey (HSD) multiple comparison test. All statistical analyses were performed using the XLSTAT software (version 2022, Addinsoft, Paris, France).

## 3. Results and discussion

*3.1. Oleoresin production*

*3.1.1. Effect of site, tree diameter and tree density*

An ANOVA analysis revealed a highly significant difference in oleoresin production between the two sites, Chareneuve and Vern at a confidence level of 95 % (p-value < 0.001). The mean oleoresin yield at Chareneuve was 4.00 kg per tree (confidence interval


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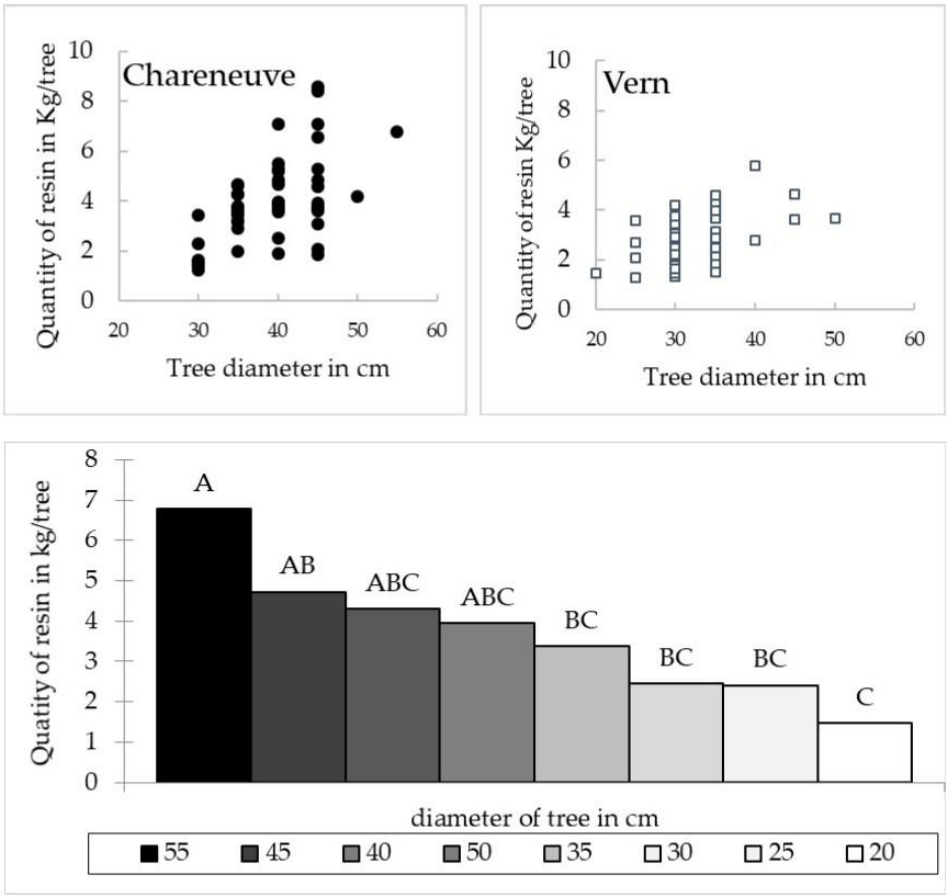


**Figure 1.** Quantity of oleoresin in kg per tree depending on tree diameter for site Chareneuve (•) and Vern (□) and for both sites. Different letters signified significative difference at confident level 95%.

(CI) between 3.62 and 4.39 kg per tree), compared to 2.79 kg per tree for Vern (CI between 2.41 and 3.17 kg per tree). Similarly, the average diameter of trees differed significantly between the two sites, with a value of 39.2 cm for Chareneuve trees and 31.9 cm for Vern ($F = 44.07$, p-value < 0.001%). At the former site, the most common tree diameter ranged between 45 and 35 cm with average oleoresin yields exceeding 3.6 kg per tree (Fig. 1). In contrast, Vern trees were characterized by lower yields (< 3.0 kg per tree) and smaller diameters, typically between 30 and 35 cm (Fig.1). However, when data from both sites were pooled, oleoresin production appeared similar for trees with the same diameter, regardless of location. Although ANOVA analysis identified three homogenous groups (A, B and C), some overlap between groups was evident because some diameters were weakly represented (Fig. 1). This may be attributed to the high variability in oleoresin production at the Chareneuve station, where trees produced high (8.0 kg) and low (1.2 kg) quantities. This difference in yield seemed less marked for the Vern station, characterized by more homogenous trees in terms of diameter. A comparison between two Spanish sites showed only minor differences in oleoresin production ranging from 2.8 kg to 3.1 kg per tree each year [6]. In Spain, Arrabal et al. [4] identified in 26 areas, some trees called "plus trees" because produced 1.5 times more than others in the same location, a ratio also observed both between and within the Cévennes sites in this study. A more recent study that compared multiple production sites in Spain, as well as the production per year and the tapping method showed that the most influential factor was the production year [2].

In general, across *Pinus* species, larger tree diameters typically have wider vertical and radial ducts, are associated with higher oleoresin yields [1, 20]. Other


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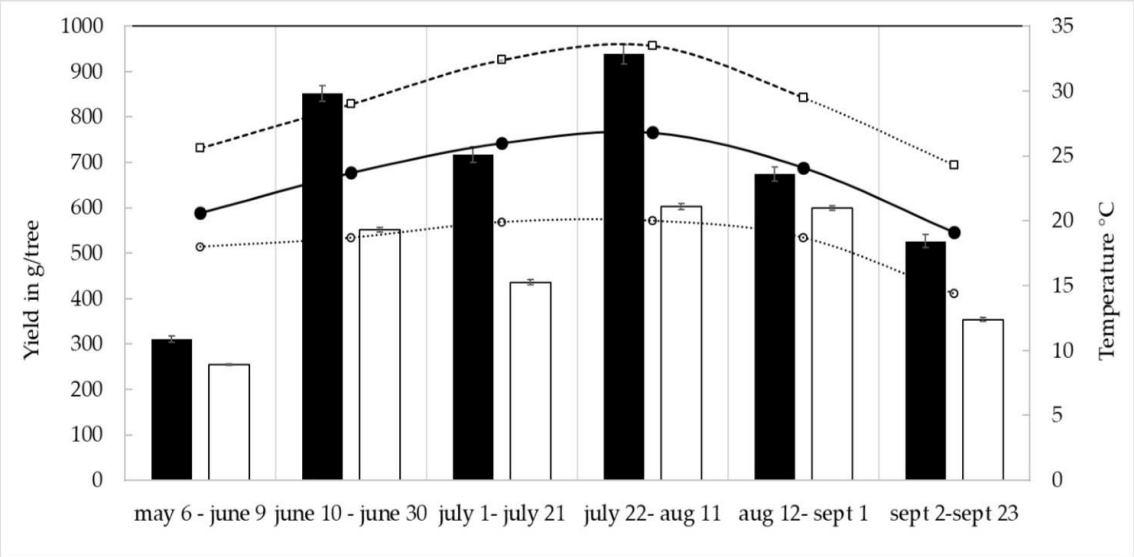


**Figure 2.** Oleoresin yield per station (Chareneuve black and Vern white) between two pikes and corresponding temperatures during the tapping season: average (•), lower (◦) and higher (□).

factors, such as viscosity and exudation pressure may, also influence the resin flow in the duct. Nevertheless, a study on *Pinus pinaster* suggested that duct characteristics did not fully explain yield variability [34].

Another potential determinant factor could be tree density. At Vern, the number of stems per hectare was double that compared to Chareneuve, suggesting high competition between the trees for light and soil nutriments and more limited development of trees. Previous studies have demonstrated a correlation between high yield and low tree densities [6, 34].

In the Landes region, the most productive trees are typically the oldest, with large diameters and well-developed crowns in low-density stands [17]. Zas et al. [35], have also found in the Atlantic station of Spain that the tree age was the main factor driving the resin production but also that slender trees produced less resin than stubby. Although the age of trees was not known, we can assume that it is correlated with tree diameter explaining, the weaker productivity of Vern. Accordingly, the Vern site was planted, in 1986, with cedars and *Pinus niger* laricio, with maritime pines regenerated naturally afterward. At Chareneuve, according to the Landowner who settled in 1977, the site was previously farmland, and the maritime pines regenerated naturally, reaching a height around 5 m in 1977. Thus, the lower productivity of trees tapped in the Vern station may be due to their smaller size (diameter and height), younger age and high density, which provoke weaker slenderness. In contrast, a comparison with another species *Pinaster pinea* of younger and smaller trees and growing at a higher density than *Pinus pinaster*, showed higher resin production for the former [27]. It is important to stress that species and resistance to stress and pests, as well as altitude, soil composition and exposure can play a part in the tree growth and influence the oleoresin yield [9, 24, 27]. In short, trees at both sites were suitable for tapping, which is recommended only for trees with a diameter greater than 20-25 cm and because yield exceeded 2 kg per tree during the harvest period [1].

*3.1.2. Effect of climatic conditions on production*

The two sites are located only 6 km apart, and therefore experience similar climatic conditions. Temperature and humidity data were obtained from *SAFRAN* climatic data, provided by Météo-France and accessed via the SICLIMA platform developed by AgroClim-INRAE [36]. For both sites, oleoresin production appeared to be affected by temperature variation. During the warmest months, June, July and August, temperatures approached or exceeded 30°C, and the total production from the 50 tapped trees ranged from 700 to 900 g at Chareneuve and 450 to 600 g at Vern. This corresponds to a daily yield of 34


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**Table 2.** Yield, density and refractive index of Eos.

| Characteristics | Chareneuve | | Vern | |
|---|---|---|---|---|
| | Hydro-distillation | Vacuum distillation | Hydro-distillation | Vacuum distillation |
| EO Yield % | 32.0[a] (1.6) | 31.2[a] (1.5) | 33.6[a] (1.7) | 32.5[a] (1.3) |
| Relative density at 20°C | 0.8626[b] (0.0002) | 0.8614[a] (0.0002) | 0.8653[d] (0.0003) | 0.8637[c] (0.0003) |
| Refractive index at 20°C | 1.4683[a] (0.0002) | 1.4681[a] (0.0003) | 1.4691[b] (0.0003) | 1.4670[b] (0.0001) |

to 44 g/tree for Chareneuve against 20-28 g/tree for Vern (Fig. 2). As temperature decreased in September, a significant reduction was observed. However, production remained higher than may despite the lower minimum temperature (14°C in September compared to 18°C in May). The rain rate was higher in September (25 mm) than in May (around 8 mm) but no correlation between this factor and yield found. This suggests a delayed flow of oleoresin after the first spike, as daily yields per tree ranged between 7.1 and 9.5 g depending on the site in May, while they exceeded 16 g in September, even for the less productive site (Vern). Accordingly, in northern Spain, the extraction yield increased from the start of the campaign for 1 month, remained stable for months and then decreased until the end of the season. The cumulative resin yield each tree followed a sigmoidal curve. This pattern was observed when a mechanized extraction method was used and can be modelled to predict the yield and managed productivity [9, 31]. In Portugal, resin production increased from April to August and then decreased, with a decrease recorded during the period of lower soil water availability [27]. These authors highlighted that the resin tapping season coincides with the growing season, from spring to autumn but, that the growth decreased when resin production increased in relation to a potential trade off in carbon allocation.

Low temperatures may reduce oleoresin synthesis and increase the relative resin viscosity, thereby reducing resin flow [10]. Rodriguez-Garcia et al. [6], reported a positive correlation between temperature and yields for *Pinus pinaster* growing in two regions with Atlantic-climate in Spain. However, in this study, the trees were located in the Cévennes, which are subject to a Mediterranean climate. The Mediterranean climate is sharply defined by high temperatures associated with frequent hot days and rare cold ones. Inter-annual temperature amplitude between January and July is high (+17 °C) [29]. Low rainfall and a high ratio of autonomous and summer precipitation are characteristics of this climate. In contrast, the Atlantic climate is characterized by abundant annual precipitation with a smaller inter-annual temperature amplitude between January and July (+13°C). The number of hot and cold days are rare. In the province of Segovia in Spain, an area with a Mediterranean climate, the most productive year was considered as relatively hot and dry, highlighting the importance of climatic variation for the same location [2]. In all cases, the highest oleoresin yields were recorded during the warmest period when the temperature exceed at 28 °C in a day.

*3.1.3. Essential oil yield, density, refractive index and composition*

First, the amount volatile compounds in the oleoresin was estimated using a rapid heating method at 150 °C, proposed by Rubini et al. [11]: values of 31.8 ± 1.5% and 32.2 ± 1.4 % were obtained for Chareneuve and Vern, respectively. Then, the suitability of both distillation methods (hydro-distillation and distillation under vacuum) was tested following the optimized conditions established in a previous research [29]. EO yields exceeded 30%, regardless of the method used, confirming the presence of a high amount of a volatile compound in the oleoresin. The yields were comparable between the 2 sites (Table 2) indicating that neither the station nor the size of the trees had a significant impact. When compared with the yield obtained from an oleoresin sample harvested in the Landes, purchased by Holiste and subjected to hydro-distillation, their value (28.7 ± 1.5%) was slightly lower than the same order of magnitude. Rubini et al. [11] reported an average EO yield of 34.25% for resin collected in Landes under similar harvesting and rapid method conditions, although yields varied widely from 15% to 52.8%. The comparison between 5 stations in Spain monitoring


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**Table 3.** Percentage of the major compounds identified in Eos.

| Compounds (%) | RT (min) | LRI experimental | Chareneuve | | Vern | |
|---|---|---|---|---|---|---|
| | | | Hydro-distillation | Vacuum distillation | Hydro-distillation | Vacuum distillation |
| α-pinene | 3.13 | 1014 | 81.4$^{aB}$ | 82.0$^{aB}$ | 75.1$^{aA}$ | 74.4$^{aA}$ |
| camphene | 3.76 | 1046 | 0.82$^{bB}$ | 0.72$^{aA}$ | 0.75$^{aA}$ | 0.74$^{aA}$ |
| β-pinene | 4.63 | 1092 | 12.1 $^{aA}$ | 13.2$^{aA}$ | 18.8$^{aB}$ | 20.4$^{aB}$ |
| δ-3-carene | 5.69 | 1132 | 0.03$^{aB}$ | 0.04$^{aB}$ | 0.01$^{aA}$ | 0.02$^{aA}$ |
| β-myrcene | 6.37 | 1154 | 1.2$^{aA}$ | 1.06$^{aA}$ | 1.22$^{aA}$ | 1.17$^{aA}$ |
| D-limonene | 7.24 | 1181 | 1.25 $^{aA}$ | 1.17$^{aA}$ | 1.11$^{aA}$ | 1.05$^{aA}$ |
| β-phellandrene | 7.5 | 1186 | 0.53$^{aA}$ | 0.55$^{aA}$ | 0.70$^{aB}$ | 0.72$^{aB}$ |
| α-terpinolene | 10.53 | 1261 | 0.23$^{aA}$ | 0.27$^{aA}$ | 0.26$^{aA}$ | 0.27$^{aA}$ |
| longifolene | 24.34 | 1519 | 1.49$^{bD}$ | 0.59$^{aB}$ | 1.02$^{bC}$ | 0.23$^{aA}$ |
| (E)β-caryophyllene | 26.25 | 1553 | 0.92$^{aB}$ | 0.39$^{bA}$ | 1.17$^{aC}$ | 0.37$^{bA}$ |
| β-caryophyllene oxide | 35.37 | 1942 | 0.02$^{bB}$ | 0.001$^{aA}$ | 0.01$^{bB}$ | 0.003$^{aA}$ |
| ***Total compounds*** | | | ***100*** | ***100*** | ***100*** | ***100*** |
| ***Compound classes*** | | | | | | |
| *Monoterpenes* | | | 97.7$^{aA}$ | 99$^{aA}$ | 97.8$^{aA}$ | 99.4$^{aA}$ |
| *Pinenes* | | | 93.5$^{aA}$ | 95.2$^{aA}$ | 93.8 $^{aA}$ | 94.8$^{aA}$ |
| *Sesquiterpenes* | | | 2.3$^{bC}$ | 1$^{aB}$ | 2.2$^{bC}$ | 0.6$^{aA}$ |
| *Ratio α/β pinene* | | | 6.7 | 6.2 | 4.0 | 3.6 |

RT : retention time, LRI experimental linear retention index.

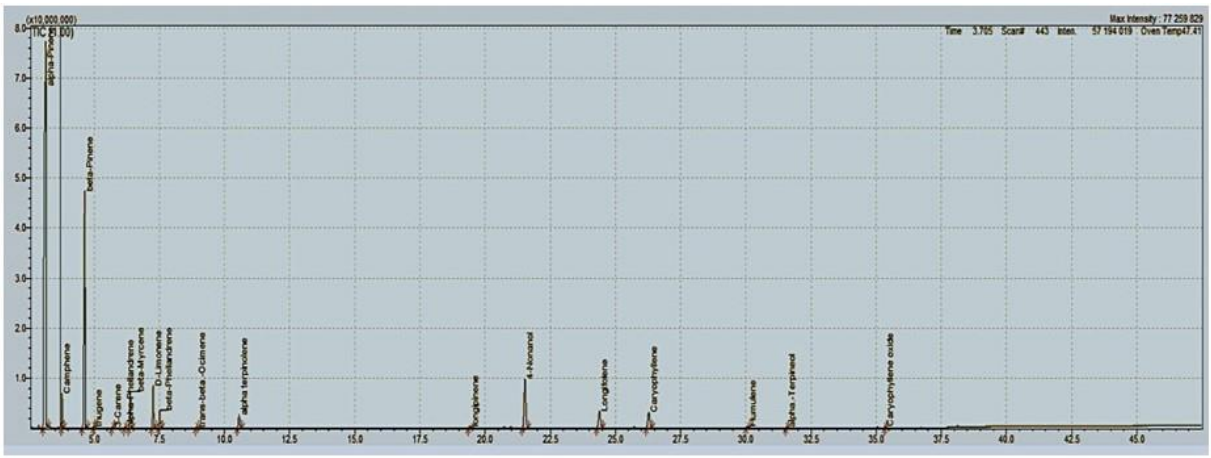


**Figure 3.** TIC of Essential oil obtained after hydro-distillation of resin from Chareneuve station.

150 trees of the same age (33 years old) showed limited variation in EO yields ranging between 27.7 and 30.3% and aligning with our findings [25].

The composition of turpentine essential oil was characterized by measuring the relative density and refractive index. According to the ISO standard (11020:1998) [30], the relative density at 20°C should range between 0.860 and 0.872 and the refractive index between 1.465 and 1.475. As shown in Table 2, the essential oils met these specifications regardless of the distillation method. For the Landes sample, the values were similar to those found for Cévennes, with a relative density of 0.8615 ± 0.0004 and a refractive index of 1.4683 ± 0.0002.

A slight difference attributable to the distillation method (Table 2) was observed, as previously reported [29]. The relative density and refractive index values were significantly lower for Chareneuve compared to Vern. This may be linked to the variation in the chemical composition of the two essential oils, as observed in Table 3 which focused on the percentage of major compounds. An example of a TIC


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**Table 4.** Acid index of rosin and kg of acid expressed in abietic acid in kg/kg depending on distillation and location.

| Station | Chareneuve | | Vern | |
|---|---|---|---|---|
| **Method** | **Hydro-distillation** | **Vacuum distillation** | **Hydro-distillation** | **Vacuum distillation** |
| Acid Index (mg KOH/g) | $150.7^{a}\pm 1.3$ | $149.5^{a}\pm 0.6$ | $154.3^{a}\pm 1.76$ | $150.17^{a}\pm 0.6$ |
| Acid content in kg/kg of rosin | $0.806^{a}\pm 0.011$ | $0.801^{a}\pm 0.013$ | $0.830^{b}\pm 0.009$ | $0.808^{a}\pm 0.001$ |

profile obtained for a turpentine EO sample is presented in Fig. 3. Although the qualitative compositions were similar between the EOs from the two sites, Vern EO presented a higher proportion of β-pinene and a lower proportion of α-pinene than Chareneuve (Table 3). Specifically, α- and β-pinene accounted for approximately 82% and 13% in Chareneuve, and 75% and 19% in Vern, respectively. Consequently, the α/β-pinene ratio was significantly higher in Chareneuve (around 6.7) compared to Vern (around 4).

Despite this difference, the proportions of each pinene are consistent with values reported for various trees (n = 94) in the Landes region [8] and with values (α- and β-pinene accounted for approximately 82% and 12.7% with a ratio of 6.4) found in the Landes of the analyzed sample in this study.

No significant difference in the total pinene content was observed between the two sites, suggesting a specificity of pinene synthase activity. Dia et al. [37], reported the presence of multiple terpene synthases in *Pinus elliottii*, including two α-pinene synthases responsible for producing between 47% and 67% of α-pinene, and one β-pinene synthase producing up to 79.95% of β-pinene. These findings indicate that the presence and expression of specific synthases largely determine the oleoresin composition. De Oliveira Junkes et al. [38] as well as Yi et al. [20]., also showed that in *Pinus elliottii* (or slash pine), individuals with high resin yields exhibited elevated β-pinene synthase gene expression, whereas those with higher α-pinene synthase expression tended to produce less resin. In our study on *Pinus pinaster*, lower resin production was associated with higher β-pinene concentrations and potentially elevated β-pinene synthase gene.

Rubini et al. [8, 11] further demonstrated that the α/β-pinene ratio can vary depending on species, tapping method, and tree provenance. For example, *Pinus pinaster* from Spain exhibited a ratio of 5 (calculated from oleoresin analysis and not from EO composition as in this study), whereas trees from Portugal showed a ratio of 2. In the Landes region of France, the ratio was around 3 under tapping conditions similar to those used in this study, but calculated considering oleoresin [8, 39]. The difference in sample nature (oleoresin against turpentine EO), in climate (Atlantic vs. Mediterranean) and tree age may contribute to the variation observed between the two regions. In southwestern Corsica and Provence, wood samples showed pinene ratios ranging from 3.3 to 10 [22].

The high β-pinene content of Vern EO may influence its aroma profile. β-pinene is known for its slightly cooling, camphoraceous character with fresh herbal notes, reminiscent of parsley, basil, or dill. In contrast, α-pinene contributes sweet, pine-like, fresh, camphoraceous, woody, and earthy nuances (source: www.thegoodscentscompany.com). Additionally, α- and β-pinene exhibit distinct medicinal properties. β-pinene is less cytotoxic than α-pinene [40] while α-pinene has demonstrated strong anti-inflammatory activity [41].

Delta-3-carene was slightly more abundant in Chareneuve EO, although its concentration remained low. Low quantities were also found in the samples from Landes. Other monoterpenes were present at similar levels across both EOs and distillation methods, except for β-phellandrene, which was slightly less abundant in Chareneuve. Environmental factors, such as light and temperature, may further influence the synthesis of specific terpenes [23].

Sesquiterpene levels were low and comparable between the two EOs, consistent with their typically low abundance in wood-derived essential oils [42]. As previously stated, their recovery enhanced by the presence of water and/or high temperatures, and hydro-distillation yielded higher sesquiterpene percentages [29]. Longifolene content was higher in Chareneuve EO, regardless of the distillation method


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**Table 5.** Rosin composition (%) depending on site.

| Site | Chareneuve | | Vern | |
|---|---|---|---|---|
| **Compounds** | **Total compounds (%)** | **Total diterpenic acids (%)** | **Total compounds (%)** | **Total diterpenic acids (%)** |
| Monoterpenes: D-limonene, $\alpha$-terpinolene | 0.51[b] | | 0.36[a] | |
| Sesquiterpenic compounds longifolene, caryophyllene humulene | 10.71[b] | | 7.61[a] | |
| Oxygenated Monoterpenes pinocarveol, cis verbenol, borneol | 0.39[b] | | <0.1 [a] | |
| Oxygenated Sesquiterpenes cubebol, longiborneol, kaurenol, kaurenal, isopimaral, caryophyllene oxide | 2.9 [b] | | 2.19 [a] | |
| Palmitic and Stearic acid | 1.13 [a] | | 2.10 [b] | |
| Sandaracopimaric acid | 0.56 [a] | 0.66 | 0.51 [a] | 0.58 |
| Pimaric acid | 6.78 [a] | 7.36 | 6.02 [a] | 6.82 |
| Levopimaric acid | 4.94[a] | 5.85 | 4.44 [a] | 5.0 |
| Palustric acid | 9.88[a] | 10.53 | 9.62[a] | 10.9 |
| Isopimaric acid | 8.21[a] | 9.73 | 7.16[b] | 8.1 |
| Abietic/dehydroabietic acid | 36.2[a] | 42.9 | 39.1 [b] | 45.1 |
| Neoabietic acid | 11.89 [b] | 12.9 | 11.1[a] | 12.6 |
| Unidentified compounds | 8.46[a] | 10.07 | 9.08 [b] | 10.9 |
| ***Total diterpenic acids*** | ***78.5*** | ***100*** | ***77.0*** | ***100*** |

(Table 3). A greater amount of $\alpha$-longipinene was also detected in Chareneuve compared to Vern. These two sesquiterpenes are often co-produced, potentially via the same biosynthetic pathway [42]. In the sample from the Landes, the percentage of caryophyllene was higher (around 1.8%), compared to Cévenes, and longifolene remained low (0.28%). Among the eleven identified (Fig. 3) and quantified compounds (Table 3), six compounds (α-pinene, β-pinene, δ-3-carene, β-phellandrene, longifolene and β-caryophyllene) allowed differentiation between the two sites. Numerous other volatile compounds are present in the EOs at low levels (data not shown), as reported in previous study [30] and some may participate in the differentiation between both EOs.

*3.2. Rosin characterization*

To compare the resins extracted from the two stations, the residual fraction obtained after distillation, i.e the rosin was collected. The rosin was cooled and analyzed without any additional heat treatment beyond the applied distillation. First, the acid index of rosin was measured, and the amount of diterpene acids was calculated (Table 4).

The acid index values showed no significant difference between the Vern and Chareneuve stations for the vacuum distillation process or between the methods using resin at the Chareneuve station (Table 4). A slightly higher value was observed for the rosin from Vern and after hydro-distillation likely due to more efficient EO removal (Table 2). Typically, the acid value ranges from 150 to 180 mg KOH/g per sample, corresponding to 0.80 and 0.90 kg/kg of diterpenic acid [1, 43].

The values obtained in this study were at the lower end of this range, possibly due to the shorter distillation time used in vacuum distillation, which may leave residual volatiles (Table 5). For comparison, a rosin sample from the Landes region sold by Biogemme had a significantly higher acid index of 161.7 ± 2.32 mg KOH/g, corresponding to 0.87kg of acid per kg of rosin. For a sample of resin collected in Landes and provided by Biogemme too, the acid index of rosin after hydro-distillation in our laboratory was found around 150 mg KOH/g. All these results suggested that a no-negligible amount of volatile are always presents in the rosin after the EO distillation and their elimination may require heating or stronger vacuum conditions or longer time.

Regarding the rosin composition (Table 5), volatile compounds (mono and diterpenes) with low boiling


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points represented 14.5% and 10.2% for Chareneuve and Vern, respectively, indicating that the vacuum distillation used did not fully collect them. Identified diterpenic acids accounted for approximately 78.5 % consistent with acid content, considering the presence of fatty acids (1.1% and 2.1%) and the fact that unidentified compounds may also diterpenic acids. The most abundant acids were abietic and dehydroabietic acids which were hardly separated using the GC method and column chromatography. These were followed by neoabietic, palustric and isopimaric acids.

Rosin is generally composed of 90 wt% diterpenic acids, with the remaining 10 wt% consisting of esters, alcohols, aldehydes and hydrocarbons [1, 8, 43]. The diterpenic fraction is mostly characterized by two acid groups, abietic and pimaric; each containing numerous isomers. The abietic group includes abietic, palustric, levopimaric, dehydroabietic, neoabietic and argyrophilic acids, which have conjugated bounds and are prone to oxidation reactions [44]. They represented the major part of the acids (around 73%) whatever at all sites (Table 5). The acids of the pimaric group (pimaric, isopimaric and sandacopimaric) were less abundant (15.5 to 17.75 %). Other acids, isomers, or hydroxylated derived compounds were also present in the rosin, but they were not clearly identified. Some more volatile compounds, such as borneol, pinocarveol, cis-verbenol and longiborneol, a sesquiterpene oxide were less abundant in the rosin of Vern compared to Chareneuve. Moreover, rosin from Chareneuve trees was characterized by a lower level of fatty acids, abietic acid and a slightly higher rate of acids with a pimarane-type skeleton (17.75 compared to 15.5%), particularly isopimaric acid.

The difference between the two rosins was less pronounced than the EOs, which may partly be due to the difficulty for the identification of some acid deriveds. Moreover, abietane-type acids can be isomerized when heated during distillation Levopimaric acid is most often isomerized to palustric acid, which then forms abietic acid and finally neoabietic acid [45]. Moreover, heating can also accelerate the oxidation of abietic acid which can be partially avoided when distillation is carried out under vacuum as in this case. An experiment involving heating rosin at 95 °C for 30, 60, 90 and 120 min showed that residual volatile compounds disappeared and that the rosin became more yellow and translucent, in agreement with previous observations [10]. The acid profile showed a decrease of levopimaric acid in favor of palustric acid, followed by abietic acid (data not shown).

In their study Pinrheiro et al. [45] demonstrated that the profile of rosin in Portugal was highly homogeneous across seven different sitescovering the Iberian region and during various campaigns. Rosin is predominantly composed of abietane acids, particularly abietic and dehydroabietic acids. A comparative analysis of the resin composition of *Pinus pinaster* from 5 regions of Spain revealed that the most abundant acids, were palustric and levopimaric acids followed by neoabietic and abietic acids, all of which share an abietane skeleton. However, differentiation between provenance was possible based on the amount of isopimaric acid and $\alpha$-pinene [25]. The Atlantic was the most distinct group, exhibiting a lower amount of isopimaric acid compared to the Mediterranean group. A broader comparison between Spain, France and Portugal highlighted isopimaric acid and pimarinal as key chemical markers associated with the geographical origin of the oleoresin [11]. Indeed, oleoresin from trees growing in southwestern France and subjected to the Atlantic climate displayed an acidic profile, with isopimaric acid being the most abundant [11]. In contrast, the rosin sample from the Landes that we distilled and analyzed was rich in acids with an abietane skeleton (44% of abietic acid and dehydroabietic acid) and the sample of rosin furnished by Holiste was also characterized by the predominance of abietane-type acid (54% of abietic acid and dehydroabietic acid). This discrepancy suggests that the chemical composition of *Pinus pinaster* oleoresin may vary significantly depending on environmental conditions, even within the same geographical region. In a recent study, Rubini et al. [46] used a multi-method approach and statistical analysis to differentiate gum rosin samples depending on Pinus species, tapping methods and geographical origins. They demonstrated that a combination of data and key factors, such as diterpenes, resin acids, acid number


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and NIR fingerprint, were determinant of the quality discrimination of rosin. However, these authors analyzed resin samples from trees, and our study, based on rosin samples after distillation, highlights the importance of analyzing both rosin and raw oleoresin to fully understand the influence of the process on the diterpenic acid profile. To the best of our knowledge, heating treatment has not been described as promoting the formation of abietane skeletons from pimarane-type acids. A preliminary study of the oleoresin composition collected at the Vern site showed that abietane-type acid was also predominant before distillation (data not shown). Given that the *Pinus pinaster* trees in the Cévennes originate from the Landes, this suggests that climate may exert a significant influence on diterpenic acid composition.

## 4. Conclusions

This study compared the productivity and composition of *Pinus pinaster* oleoresin harvested from two sites in the Cévennes, a region in southeast of France. The tapping campaign began in May and continued for five months. Oleoresin production was higher during the warmest months (June July and August) when the temperature was exceeded 28°C. The diameter of trees and density of stems per hectare have been identified as parameters that influence productivity. Indeed, the Vern site, characterized by the smallest tree diameter (32 cm against 39 cm for Chareneuve) and the highest density of stems 1,600 against 800 for Chareneuve) exhibited a significant lower oleoresin production (2.79 kg per tree against 4 kg per tree). The turpentine EO yield (approximately 30%) obtained after distillation was similar across both sites and aligned with other sites in France (Les Landes) or European area such as Spain and Portugal. However, quantitative differences in EO composition were observed between the Vern and Chareneuve sites. Notably, a higher content of β-pinene was found in the EO obtained from the Vern station (19% against 12% for Chareneuve station) which could be associated with the lower oleoresin productivity. This compound, along with its specific synthase, may serve as a genetic marker to detect certain growth conditions.

Regarding the rosin characteristics, both sites exhibited relatively a low acid index (around 150 mg of KOH per g), explained by the absence of rosin post-heating treatment after distillation. The diterpenic acid profiles were dominated by abietane-type acid skeletons such as abietic and dehydroabietic acids. This specific composition is characteristic of oleoresins tapped in Mediterranean climate regions. This contrasts with the Landes region, where pimarane-type acids are more prevalent, reinforcing the role of climate in shaping the rosin composition. Interestingly, isopimaric acid was a distinguishing component between the two Cévennes sites. These findings warrant further investigation across additional site types in the Cévennes to better understand the adaptability of *Pinus pinaster* to Mediterranean climates and soils. Moreover, the observed regional variability in the composition of both oleoresin by-products needs to be investigated further and may represent a valuable asset for the valorization of non-timber pine products.

## Disclaimer (artificial intelligence)

Author(s) hereby state that no generative AI tools such as Large Language Models (ChatGPT, COPILOT, etc.) and text-to-image generators were utilized in the preparation or editing of this manuscript.

## Authors' contributions

Performed the choice and characteristic of the two location, supervised the tapping campaign and analyzed the data of resin production and revised the article, V.G., M.L.; performed the analyzes, the data acquisition and their interpretation, A.B., L.S.; conceived and designed the study, chosen the methodology, validated the results and contributed in writing the manuscript and revised, P.C., P.R.

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## Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

## Conflicts of interest

The authors declare that they have no conflict of interests.

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