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Sources of variance in the volatile contribution of juniper to gin

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Abstract

Why was the work done: Juniper berries and other plant botanicals are used in the production of beverages and contribute signature flavour and aroma. Inconsistent or inferior quality of botanicals is a concern and result in consumer dissatisfaction, and reduced sales.
How was the work done: The impact on gin quality of the source and harvest year of juniper berries was investigated by analysis of distillates using Gas Chromatography - Mass Spectroscopy with Solid Phase Micro Extraction (GCMS-SPME).
What are the main findings: Regional variation and drying regimes were found to impact on the concentration of volatiles and their profile. The least soluble compounds were most affected by post-harvest drying and increased in abundance whereas the more soluble compounds decreased in abundance.
Why is the work important: This work will be of interest to distillers, suppliers of botanicals and juniper farmers, and will inform drying regimes consequent on climate change.

Keywords

gin, juniper, quality, distillates, analysis, volatiles

Introduction

The luxury food industry recognises the importance of a passion for quality, innovation, and authenticity (Lingham 2022). Within the premium sector of the distilled spirits industry, the origin of ingredients is a defining feature of the product. For producers, a ready and consistent supply of ingredients is important to limit variation between batch to batch, year to year, and brand to brand (Berning et al. 2017). Consistency is important as it allows the product to establish a specific flavour profile with consumers expecting 'house style' characteristics of the product (Soltoft 1974). There has been a considerable expansion of small producers ('craft') of gin leading to a boom in sales over the past decade (Black 2022). Consequently, increasing juniper sales highlights the importance of consistency and quality of supply. In the UK gin market, the largely overseas juniper harvest is presented to the industry in November, where samples are considered and compared with the current batch.

The characteristics of juniper are key, as EU regulation 2019/787 defines gin as 'a juniper-flavoured spirit drink produced by flavouring ethyl alcohol of agricultural origin with juniper berries (*Juniperus communis* L.)', with distilled gin made of natural botanicals 'provided that the juniper taste is predominant' (European Commission 2019).

The same species of hop grown in different parts of the world are reported to contain different volatile compounds (Van Holle et al. 2017; Rodolfi et al. 2019; Lafontaine et al. 2021). Many of these are produced by the highly conserved mevalonate pathway which is also found in the berries of the juniper plant (Verma and Shukla 2015). Regionality of plant characteristics is termed 'terroir' in viticulture (Van Leeuwen and Seguin 2006), leading to region-to-region differences in secondary metabolites and flavour compounds. So profound is this, that regions are designated protected areas of origin (e.g. Champagne and Bordeaux) (Haeck et al. 2019). The concept of 'vintage' is also found within viticulture and considers harvest to harvest variation between years (Roullier-Gall et al. 2014). The origins of these variations are multiple, including latitude (Lafontaine et al. 2021), mineral content of soil, and average rainfall (Rodolfi et al. 2019).

Differences have been reported the in volatile content of juniper berries using pentanol extraction (Pažitná and Špánik 2014), volatile content of needles in relation to latitude (Martz et al. 2009) with hexane extraction, and variations of essential oil content based on hydro distillation (Agastra et al. 2021). However, directly transferable research to the commercial gin industry with relevant extraction profiles is limited.

In this work, the levels of terpenes (from the mevalonate pathway) found in juniper distillates were determined. Terpenes are secondary metabolites which in the juniper plant deter herbivores, attract pollinators, and defend against pathogens (Barzalona and Casanova 2008). Terpenes are a significant contribution (Vichi et al. 2005) to the flavour profile of juniper. Accordingly, Hodel et al (2019) developed a qualitive and quantitative method for terpenes and other volatiles in gin. Using this appraoch, key terpenes in juniper berries from multiple areas and harvests were analysed to determine the impact of growing region and post-harvest drying regimes on final spirit quality.

Materials and methods

Juniper berries

Juniper berries were obtained from Krautermix GmbH (Abtswind, Germany), sifted and dried to 15% (w/w) moisture at a temperature of 37°C. Berries from the wet harvest in 2017 required longer drying periods to reach the desired moisture content than the dryer harvest of 2018. The origin of and harvest year of the juniper samples are reported in Table 1.

Grain neutral spirit

Grain neutral spirit (GNS) was supplied by Hayman Kimia (Chelmsford, UK) at 96.3% alcohol by volume (ABV).

Still configuration

The laboratory still consisted of a 1L Quickfit™ round bottom glass flask, cone screw thread connector, red spirit filled thermometer, still head, Liebig condenser, elbow joint, 25mL beaker (Fisher Scientific, Loughborough, UK; Figure 1), and a 1L electrothermal heat mantle (Electrothermal, Staffordshire, UK). Water was cooled using a

Lauda circulation chiller MC600 (Lauda Technology Ltd, Stamford, UK) at 2°C.

Table 1.

Origin and year of harvest of juniper berries. Different batches harvested within the same year are from different areas within the same country.

Region	Harvest	Batch Code
Albania	2017	19120-17
Albania	2018	19240-18
Albania	2018	19230-18
Bosnia	2017	19010-17
Bosnia	2017	19020-17
Bosnia	2018	19140-18
Bosnia	2018	19130-18
Macedonia	2017	19060-17
Macedonia	2017	19050-17
Macedonia	2018	19170-18
Macedonia	2018	19180-18
Montenegro	2017	19110-17
Serbia	2017	19100-17
Serbia	2018	19210-18
Serbia	2018	19220-18
Kosovo	2017	19070-17
Kosovo	2018	19190-18
Kosovo	2018	19200-18
Italy	2017	19040-17
Italy	2017	19030-17
Italy	2018	19150-18
Italy	2018	19160-18
Kosovo	2017	19080-17

Juniper berries (8g) were added to grain neutral spirit (800 mL), diluted to 60% ABV with distilled water, and steeped for a minimum of 12 hours at ca. 20°C.

Distillation

The berries in GNS were distilled in the system described in Figure 1. To ensure industrial applicability, the distillate was split into two sections: 40 mL foreshots and a 450 mL main cut (which was used for analysis). Each juniper sample was distilled in triplicate. The flow rate of the distillate was 2 ± 0.04 mL/min.

Alcohol measurement

Density and temperature were measured using an Anton Paar DMA 35 Density Meter (Anton Paar, Graz, Austria) with the total alcohol by volume calculated using the Practical Alcohols Table (European Commission, 1978).

All samples were diluted with distilled water to 8% ABV, to give a total volume of 5 mL. An internal standard mix of a known quantity (Table 2) was used for calibration with the internal standard (3-octanol) added at 9.48 mg/L with an in-sample concentration of 2.5 mg/L.

Gas Chromatography-Mass Spectrometry with Solid Phase Micro extraction

Gas Chromatography-Mass Spectrometry was performed using an AOC 5000 autosampler coupled to a GCMS-QP2010 Ultra (Shimadzu, Kyoto, Japan) with separation performed using a HP5MS (30 m × 0.25 mm × 0.25 µm) column (Agilent, Santa Clara, CA, USA). The carrier gas was helium at a flow rate of 1.6 mL/min. Sampling required a 65 µm PDMS/DVB solid phase microextraction fibre (SPME; Supelco, Bellefonte, PA, USA). The sample was conditioned for 5 min at 50°C followed by 30 minutes extraction in the headspace of the vial and 1 min desorption into the inlet of the GC. The inlet temperature was 250°C, with an initial oven temperature of 55°C, and a 3 min hold time. The oven temperature was increased to 60°C at 1°C/min with a 1 min hold, increased to 65°C at a rate of 5°C/min, followed by an increase to 110°C at 10°C /min with a 10 min hold. The oven was then heated to 120°C at 10°C/min with a 3 min hold; increased to 250°C at 10°C/min and finally increased to 320°C at 40°C/min held for 3 min, resulting in a total time of 45.25 min. The mass spectrometer operated with an ion source temperature of 175°C and an interface temperature of 280°C. The solvent cut time was set to 2.9 min. One scan every 0.5 sec was performed between m/z 35 and 500.

Figure 1.

Configuration of laboratory still.

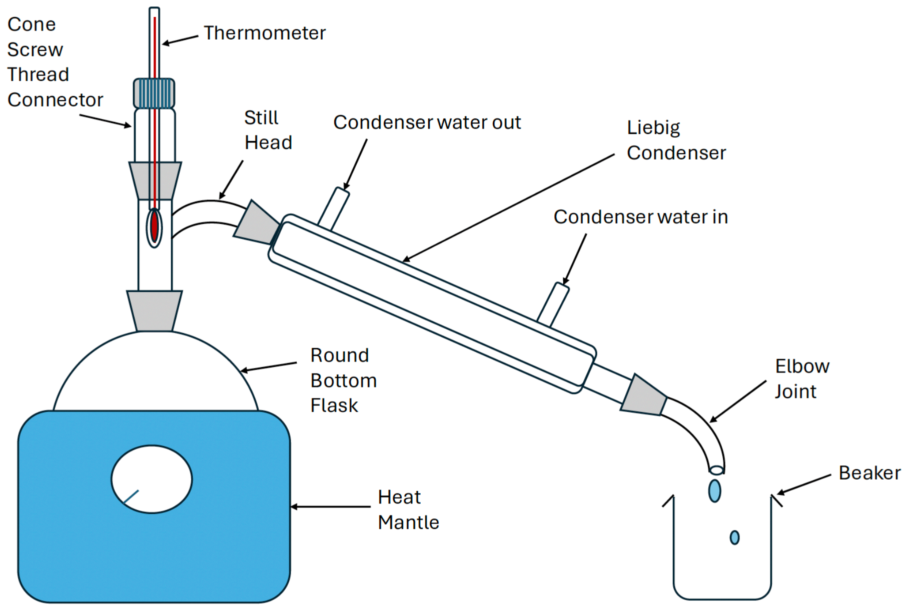


Table 2.

Purity and sensory attributes of reference standards used in calibration curves.

Compound	CAS	Group	Molecular formula	Supplier	Purity (%)	Sensory attributes
α-Pinene	7785-70-8	Monoterpene	$C_{10}H_{16}$	Sigma-Aldrich, Dorset, UK	99	Fresh Pine, Camphor, Sweet, Earthy, Woody (i)
β-Pinene	18172-67-3	Monoterpene	$C_{10}H_{16}$	Sigma-Aldrich	99	Dry Woody, Resinous Pine, Hay, Green (i)
α-Phellandrene	99-83-2	Monoterpene	$C_{10}H_{16}$	SAFC	85	Citrus, Herbal, Terpenic, Green, Woody, Pepper, Black Pepper (ii)
β-Myrcene	123-35-3	Monoterpene	$C_{10}H_{16}$	Sigma-Aldrich	AS	Minty, Terpenic (i)
R-Limonene	5989-27-5	Monoterpene	$C_{10}H_{16}$	Sigma-Aldrich	95	Peppery, Terpenic, Spicy, Balsamic, Plastic (i)
Y-Terpinene	99-85-4	Monoterpene	$C_{10}H_{16}$	FlukaÂ® Analytical, Dorset, UK	97	Orange, Citrus, Sweet (i)
Linalool	78-70-6	Oxygenated Monoterpene	$C_{10}H_{18}O$	Sigma-Aldrich	97	Sweet, Musk, Waxy, Ambrette, Dry, Woody (ii)
Terpinen-4-ol	2438-10-0	Oxygenated Monoterpene	$C_{10}H_{18}O$	FlukaÂ® Analytical,	AS	Oily Woody Terpenic (i)
β-Caryophyllene	87-44-5	Sesquiterpene	$C_{15}H_{24}$	SAFC	80	Citrus, Floral, Sweet, Blueberry (i)
α-Humulene	6753-98-6	Sesquiterpene	$C_{15}H_{24}$	Sigma-Aldrich	98	Peppery, Woody, Earthy, Musty (i)
3-Octanol (ISTD)	589-98-0	Alcohol	$C_8H_{18}O$	Sigma-Aldrich	99	n/a

Data processing

Compounds of interest were selected after preliminary work identifying compounds found within gin and juniper berries based on the highest concentration, using GC–MS SPME analysis and the NIST08s Mass Spectral Library Software (NIST, Maryland, USA).

Calculation of relative abundance

The distribution of compounds was diverse and would not allow one figure to contain all compounds for comparison. The levels were bench marked against juniper berries from Macedonia (one of the most established growing regions) as standard. The compound levels for Macedonia were established as the mid-point and levels above or below are expressed proportionately. As relative abundance it is dimensionless and has no units.

Results and discussion

Harvest year

Meteorological data (Table 3) shows that the rainfall in the harvest months for juniper - September, October and November - in seven locations. This varied, with 57% more during 2017 compared to 2018. Accordingly, juniper berries harvested in 2017 required more drying to achieve the optimum 15% moisture content, compared to berries harvested in 2018. The total volatile levels (relative abundance) across all growing regions combined was lower for juniper berries from the wet harvest of 2017, compared to those harvested in 2018 (Figure 2). Indeed, there was a 12% reduction in volatiles in the 2017 juniper samples compared with 2018.

It is an important to note that this observation relates to a difference in moisture content at harvest and not directly growing conditions. The multivariable nature of plants and other factors throughout the growing season mean that these will influence the profile of compounds in berries before harvest. Samples of juniper from more areas - both established and prospective - including multiple harvests would be an area for future study.

Closer analysis of the specific volatile compounds in berries from each harvest, show that the distribution of relative abundance is not evenly spread throughout the sample set (Table 4, Figure 2). The standard deviation for β-caryophyllene is 58.8, whilst β-pinene has a standard deviation of 23.1 (Table 4). Differences in the content in the final spirit of β-pinene will result in changes in sensory impact with woody, pine characteristics of turpentine, eucalyptus, and camphor. Similarly, variations in the concentration of β-caryophyllene impact the sensory characteristics of the finished product with dry, spicy, clove and woody.

The variations reported here could reflect differences in water solubility and volatilities of compounds when exposed to heating to reduce the moisture content, post-harvest. The process of heating drives off the least soluble volatiles first. The water solubility verses difference in relative abundance is illustrated by the results reported in Table 4. The most water-soluble compounds (terpinen-4-ol) exhibit a decrease in concentration on comparing wet to dry, whereas the least water-soluble compounds (β-caryophyllene, α-humulene) show the biggest increase.

Table 3.

Meteorological information for harvest months (September, October, November) in 2017/18 for all regions (www.meteoblue.com)

	Harvest Year		
	2017	2018	
Country	**Harvest Rainfall (mm)**		**Latitude (°N)**
Albania	212	143	41.33
Bosnia	321	157	43.85
Macedonia	80	62	41.87
Montenegro	223	116	42
Serbia	141	60	42.6
Kosovo	104	48	42.57
Italy	137	188	44.02
Total	**1218**	**774**	

All in all, the trend is for a reduction in abundance and a decrease in level after a wet harvest. The pattern observed relative to solubility and volatility suggests that the most soluble molecules are more stable and less volatile when dried. Factoring in differences in volatility, these results suggest that the solubility in water has more influence than volatility, on the impact of post-harvest processing.

Figure 2.

Relative abundance of key compounds in juniper berry distillates from the 2017 (wet) and 2018 (dry) harvests.

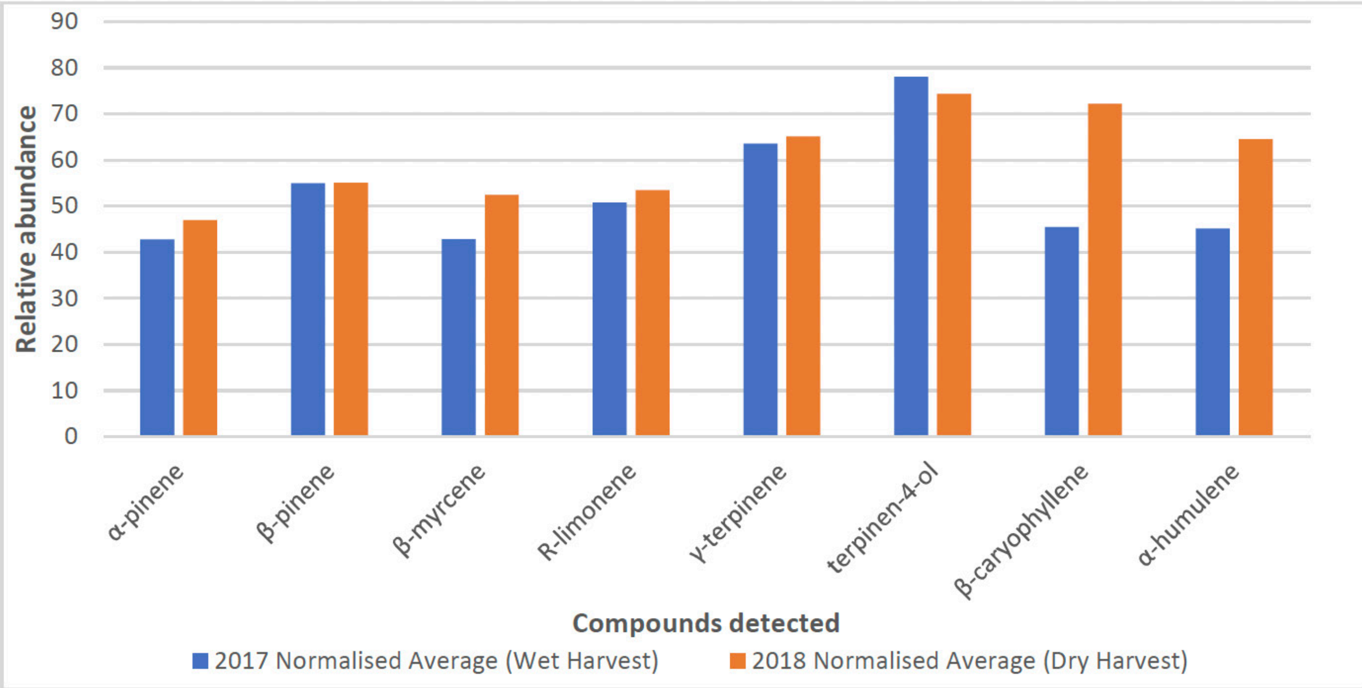


Table 4.

Key juniper compounds: comparison of vapour pressure, water solubility and percentage (%) difference between dry and wet.

Compound	Vapour Pressure (mm Hg at 20°C)	Water Solubility (mg/L)	% Difference
α-Pinene	4.75	2.49	9.8
β-Pinene	2.73	7.061	0.1
β-Myrcene	2.29	5.6	22.4
R-Limonene	0.198	13.8	5.2
γ-Terpinene	1.075	8.68	2.6
Terpinen-4-ol	0.048	386.6	-4.8
β-Caryophyllene	0.013	0.05011	58.9
α-Humulene	0.008	0.01396	43.0

Low						High

The drying of botanicals could potentially contribute to the volatile content of distilled beverages, as it may provide a preliminary volatilisation, distillation step. The drying process is required to produce stable botanicals and achieve a consistent batch to batch product. Without appropriate drying protocols there is increased risk of microbial spoilage (Chao et al. 2017) and fluctuations in moisture content could affect product consistency (Chao et al. 2017) when weighed out in the distillery.

Juniper composition by country of origin

Juniper samples from seven harvest regions were analysed for the presence of eight key marker compounds. Significant variation was noted across country of origin (Figure 3) with standard deviation ranging threefold from high (terpenin-4-ol) to low (β-myrcene) (Figure 4). However, on considering the abundance of compounds in juniper distillates by origin it is important to note similarities and differences. Figure 3 illustrates the variation of the six regions relative to that of Macedonia, with α-pinene, β-pinene and β-myrcene with limited variability possibly being resilient to regionality. Of the differences in abundance between the regions of juniper production (Figure 4), terpien-4-ol, β-caryophyllene and γ-terpinene exhibited the biggest difference in standard deviation. These compounds show high variability and may be regional indicator compounds.

The highly variable nature of plants with multiple factors and environmental influences throughout the growing season will inevitably influence the levels of compounds in berries preharvest. With an ever changing climate and changes in growing conditions for farmers of all crops, it is important to monitor the effect of these changes on existing juniper crops and those from emerging areas.

Samples of juniper berries pre- and post drying would enable future work together with samples from more geographic areas, particularly those that

Figure 3.

Abundance of eight key compounds (relative to Macedonia) found in juniper distillates by country of origin.

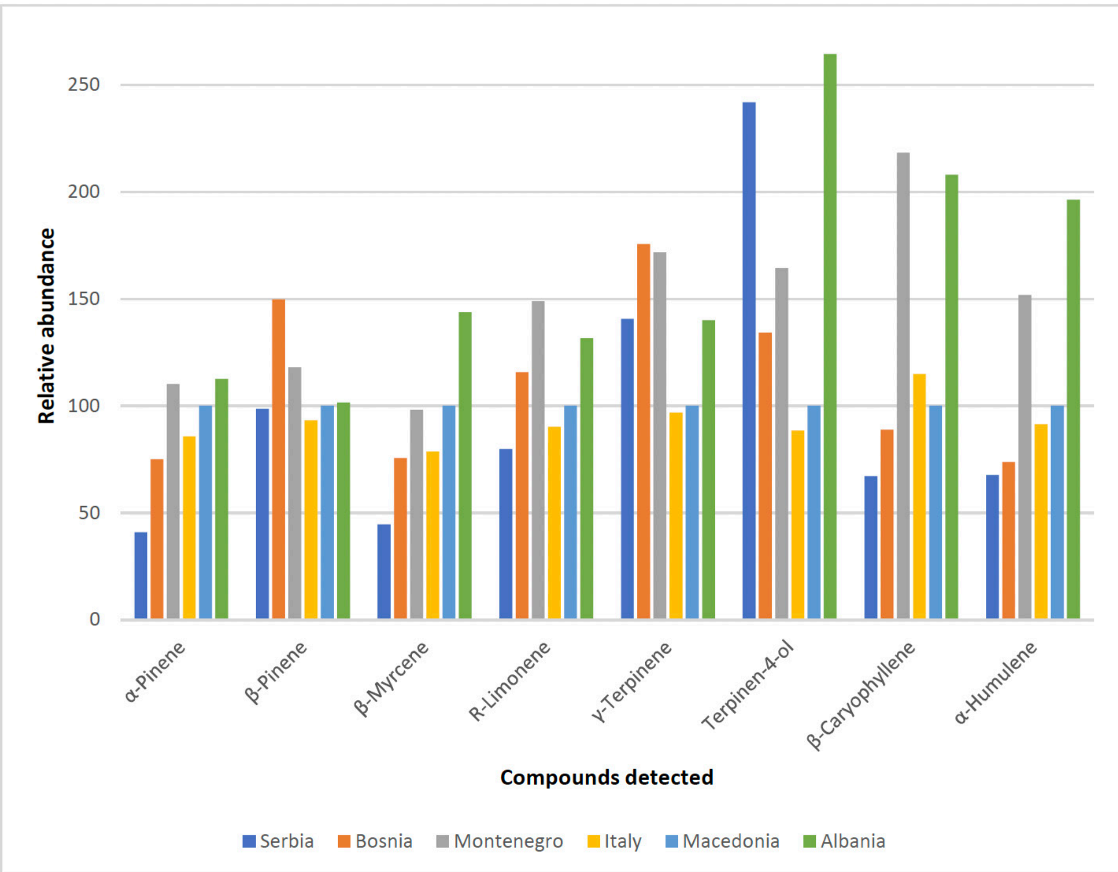
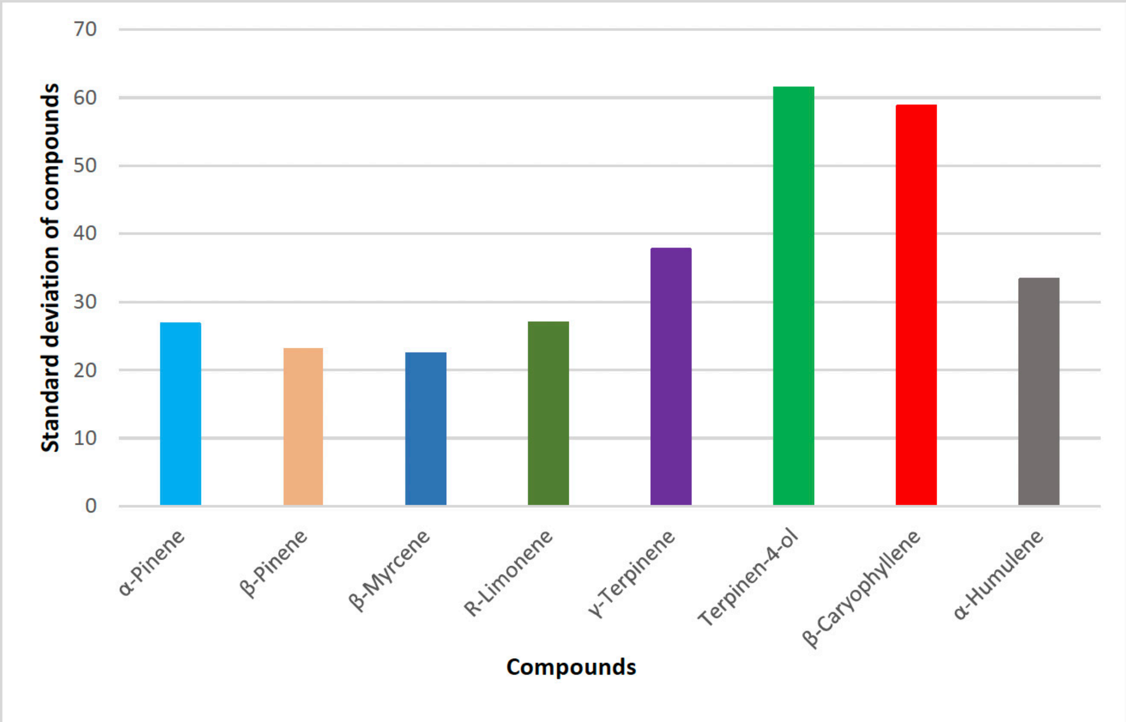


Figure 4.

Standard deviation of eight key compounds in juniper distillates between regions with combined harvests when comparing different regions by country of origin.



emerge with the shift in climate. Juniper samples from different countries of origin contained significant variation across eight marker compounds (Figure 2). The standard deviation of these compounds vary (Figure 3), suggesting these differences are not uniform across the sample set. It is important to note that this effect is observed across multiple regions with varying climates and growing conditions.

Different approaches have been used for studies on juniper berries include pre-treatment with grinding (Marković et al. 2018), soxhlet extraction with acetone (Nasri et al. 2011), super critical extraction with carbon dioxide (Sovilj et al 2011), microwave hydro distillation (Marković et al. 2019) and 'instantaneous controlled pressure-drop' (Naji et al. 2008). The common themes of all these approaches are dissolving the terpenes from the plant matter into a solvent. The method used in this study was chosen as being relevant and transferable to the commercial gin distillation process.

Conclusions

The profile of key compounds in juniper berries were measured between different harvests and different growing regions. Variations were found in the volatiles from area to area and harvest to harvest. The regional variations observed could potentially impact on the sensory characteristics of the gin in contributing key attributes including dry woody, resinous pine, hay, green, minty, and terpenic.

A review of the levels of compounds from harvest-to-harvest show that the least soluble compounds are most affected by post-harvest drying increasing in abundance whereas the most soluble compounds decrease in abundance with extended drying. Accordingly, the sensory characteristics most likely to be impacted in the final spirit include citrus, floral, sweet, peppery, woody, earthy and musty on the final spirit quality.

This information will be of interest to gin distillers and botanical farmers in making more informed decisions on the sourcing, timing of harvesting and drying regime of juniper berries.

Author contributions

Matthew Pauley: Conceptualisation, methodology, writing (original draft).
Annie Hill: Supervision, writing (review, and editing).

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Conflict of interest

The authors declare they have no known competing financial interests or personal relationships that could have appeared to influence the work in this paper.

References

Agastra A, Gixhari B, Kadiasi N, Ibraliu A. 2021. Influence of environmental factors in the composition of essential oils content of *Juniperus communis* L. berries, in Southeast part of Albania. *IJEES* 11:943-948. http://dx.doi.org/10.31407/ijees11.436

Berning J, Costanigro M, Mccullough MP. 2017. Can the craft beer industry tap into collective reputation? *Choices* 32:1–6. https://doi.org/10.22004/ag.econ.261902

Black K. 2022. Gin. p 423-440, *In* Russell I, Stewart GG, Kellershohn J (eds), *Whisky and Other Spirits*, Academic Press, Massachusetts, USA.

Chao J, Dai, Y, Cheng HY, Lam W, Cheng YC, Li K, Peng WH, Pao LH, Hsieh MT, Qin XM, Lee MS. 2017. Improving the concentrations of the active components in the herbal tea ingredient, *Uraria crinita*: The effect of post-harvest oven-drying processing. *Sci Rep* 7:38763. https://doi.org/10.1038/srep38763

European Commission. 1978. *Practical alcoholic strength tables. Volume 1.* https://op.europa.eu/en/publication-detail/-/publication/05b3e747-f169-424e-af99-9a6879fb44f3/language-en

European Commission. 2019. *Guidelines for the implementation of certain labelling provisions of Regulation (EU) 2019/787 of the European Parliament and of the Council of 17 April 2019 on the definition, description, presentation and labelling of spirit drinks, the use of the names of spirit drinks in the presentation and labelling of other foodstuffs, the protection of geographical indications for spirit drinks, the use of ethyl alcohol and distillates of agricultural origin in alcoholic beverages.* PE/72/2023/REV/1 http://data.europa.eu/eli/reg/2024/1143/oj

Haeck C, Meloni G, Swinnen J. 2019. The value of terroir: A historical analysis of the Bordeaux and Champagne geographical indications. *AEPP* 41:598–619. https://doi.org/10.1093/aepp/ppz026

Van Holle A, Van Landschoot A, Roldán-Ruiz I, Naudts D, De Keukeleire D. 2017. The brewing value of Amarillo hops *(Humulus lupulus* L.) grown in northwestern USA: A preliminary study of terroir significance. *J Inst Brew* 123:312-318. https://doi.org/10.1002/jib.433

Lafontaine S, Caffrey A, Dailey J, Varnum S, Hale A, Eichler B, Dennenlöhr J, Schubert C, Knoke L, Lerno L, Dagan L, 2021. Evaluation of variety, maturity, and farm on the concentrations of monoterpene diglycosides and hop volatile/nonvolatile composition in five *Humulus lupulus* cultivars. *J Agric Food Chem* 69:4356-4370. https://doi.org/10.1021/acs.jafc.0c07146

Van Leeuwen C, Seguin G. 2006. The concept of terroir in viticulture. *J Wine Res* 17:1-10. https://doi.org/10.1080/09571260600633135

Lingham S, Hill.I, Manning. L .2022. Artisan food production: what makes food 'artisan'? in Dana LP, Ramadani V, Palalic R, Salamzadeh A. 2022. *Artisan and Handicraft Entrepreneurs*. Springer International Publishing, New York, USA.

Marković MS, Milojević SŽ, Bošković-Vragolović NM, Pavićević VP, Babincev LM, Veljković VB. 2019. A new kinetic model for the common juniper essential oil extraction by microwave hydrodistillation. *Chin J Chem Eng* 27:605-612. https://doi.org/10.1016/j.cjche.2018.06.022

Martz F, Peltola R, Fontanay S, Duval RE, Julkunen-Tiitto R, Stark S. 2009. Effect of latitude and altitude on the terpenoid and soluble phenolic composition of juniper (*Juniperus communis*) needles and evaluation of their antibacterial activity in the boreal zone. *J Agric Food Chem* 57:9575-9584. https://doi.org/10.1021/jf902423k

Naji G, Mellouk H, Rezzouget S-A, Allaf K. 2008. Extraction of essential oils of juniper berries by instantaneous controlled pressure-drop: Improvement of DIC process and comparison with the steam distillation. *J Essent Oil-Bear Plants* 11:356–364. https://doi.org/10.1080/0972060X.2008.10643641

Nasri N, Tlili N, Elfalleh W, Cherif E, Ferchichi A, Khaldi A, Triki S. 2011. Chemical compounds from Phoenician juniper berries (*Juniperus phoenicea*). *Nat Prod Res* 25:1733-1742. https://doi.org/10.1080/14786419.2010.523827

Pažitná A, Špánik I. 2014. Enantiomeric distribution of major chiral volatile organic compounds in juniper-flavored distillates, *J Sep Sci* 37:398–403. https://doi.org/10.1002/jssc.201301151

Rodolfi M, Chiancone B, Liberatore CM, Fabbri A, Cirlini M, Ganino T. 2019. Changes in chemical profile of Cascade hop cones according to the growing area. *J Sci Food Agric* 99:6011-6019. https://doi.org/10.1002/jsfa.9876

Roullier-Gall C, Boutegrabet L, Gougeon RD, Schmitt-Kopplin P. 2014. A grape and wine chemodiversity comparison of different appellations in Burgundy: vintage vs terroir effects. *Food Chem* 152:100-107. https://doi.org/10.1016/j.foodchem.2013.11.056

Søltoft M. 1974. A profile method for sensory analysis of beer and its use in assessing flavour stability and flavour consistency. *J Inst Brew* 80:570-576. https://doi.org/10.1002/j.2050-0416.1974.tb03651.x

Sovilj MN, Nikolovski BG, Spasojević MD. 2011. Critical review of supercritical fluid extraction of selected spice plant materials. *Maced J Chem Chem Eng* 30:197-220. https://doi.org/10.20450/mjcce.2011.35

Tomi F, Barzalona M, Casanova J, Luro F. 2008. Chemical variability of the leaf oil of 113 hybrids from *Citrus clementina* (Commun) × *Citrus deliciosa* (Willow Leaf). *Flavour Fragr J* 23:152-163. https://doi.org/10.1002/ffj.1867

Verma N, Shukla S. 2015. Impact of various factors responsible for fluctuation in plant secondary metabolites, *J Appl Res Med Aromat Plants* 2:105-113. https://doi.org/10.1016/j.jarmap.2015.09.002

Vichi S, Riu-Aumatell M, Mora-Pons M, Buxaderas S, López-Tamames E. 2005. Characterization of volatiles in different dry gins. *J Agric Food Chem* 53:10154-10160. https://doi.org/10.1021/jf058121b