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Quality of the essential oil from two hop varieties introduced in Rio de Janeiro, Brazil

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Keywords: Comet, Triumph, myrcene, gas chromatography, Humulus lupulus.

Abstract

The objective of this work was to compare the essential oil yield and quality from two hops (*Humulus lupulus* L., Cannabaceae) varieties, namely Comet and Triumph cultivated in the mountain area of the state of Rio de Janeiro. The samples consisted of dry cones, obtained with local producers. The essential oils were obtained by hydrodistillation in a Clevenger apparatus. Residual humidity content was determined by azeotropic distillation with a Dean-Stark apparatus. Oil composition was evaluated by gas chromatography with flame ionization and mass spectrometry detectors. For quantitation, four replicates were distilled and analyzed after the addition of an internal standards. Identification was based in mass spectra comparison with commercial libraries and calculation of linear retention indices. For the samples tested, the Comet variety yielded 2 times more essential oil, with 4.1%, while the Triumph yield was only 2.0% (dried basis). Comet oil was rich in myrcene (64.9%) and β -caryophyllene (8.3%). The major compounds in Triumph oil were α -humulene (41.7%), myrcene (28.5%) and β -caryophyllene (11.5%). While the data from Triumph are in good agreement with literature, a higher yield for the Comet variety was recorded. For both varieties, it was verified a good adaptation of the hops to the mountain area near Rio de Janeiro.

1. Introduction

One of the 4 obligatory ingredients in beer, hop (*Humulus lupulus* L., family Cannabaceae) is a species originary from the northern hemisphere, with occurrence in Europe, North America, and Asia [1]. Although some introduction attempts have been made in Brazil during the last quarter of the 19th century, only recently this culture has reached commercial success. Several varieties have been introduced in the South of the country, as well as in the mountain areas of the states of São Paulo and Rio de Janeiro. Following the boom in the market of artisanal breweries, many local producers started cultivation of new varieties. Herein, we report the results on the quality assessment of the essential oil from two hop varieties, Comet and Triumph, cultivated in the city of Petrópolis, a mountain area near Rio de Janeiro.

2. Material and Methods

All solvents used were from HPLC grade (Tedia Brazil). The dried hop samples (*Humulus lupulus* L. var. Comet and *H. lupulus* L. var. Triumph), crop 2023, were kindly provided by a producer from the city of Petrópolis, RJ (S 22°25' 36.5", W 43°17'2.5"). Each sample (100 g) was distilled in a Clevenger-type apparatus according to the Brazilian Pharmacopoeia [2] (the same as in the European Pharmacopoeia). Oil yield was expressed in volume by weight and are expressed in dried basis. A 0.1 % solution of the oil in hexane and methyl octanoate as added as internal standard, and 1.0 μ L was injected in split mode (1:50). Oils were analyzed in an Agilent 7890B gas chromatograph (GC) fitted with flame ionization detector (FID) and using a DB-5 column (30 m x 0.25 mm x 0.25 μ m), with hydrogen as carrier gas (1.5 mL/min). Oven temperature was programmed from 60 to 240 °C, at 3 °C/min. The injector was operated at 250°C and the detector at 280 °C. For quantitation, predictive response factors were used. All calculations were performed using a series of Excel[®] pre-programmed electronic sheets [3]. The samples were also injected in an Agilent 5975C mass selective detector system, using the same column and conditions as stated for GC-FID. Helium was used as carrier gas (1.0 ml/min). Ionization energy was 70 eV, at 3.15 scans/s, from 40 to 350 u. Ion source was kept at 200 °C, mass analyzer at 150 °C and transfer line at 260 °C. For compound identification, mass spectra were compared to data from commercial libraries, and linear retention indices were calculated [4].

3. Results

The oil yield (dried basis) was 4.1 for the Comet and 2.0% for the Triumph varieties. Main constituents for the

Comet oil were myrcene (64.9%), β -caryophyllene (8.3%), α -selinene (3.7%), β -selinene (3.5%), and (*E*)- β -ocimene (1.8%). For the oil from the Triumph variety, α -humulene was the main constituent (41.7%), followed by myrcene (28.5%), β -caryophyllene (11.5%), δ -cadinene (2.1%), and humulene epoxide II (1.5%).

4. Discussion and Conclusions

The oil yield for the Triumph is slightly above the average, but its chemical profile is within the reported data for this variety [5]. The composition of the Comet variety was characteristic, with a high content of myrcene and low content of α -humulene. The yield, however, was twice (4.1%) the average for the variety. These results are quite interesting and stimulating for the hop producers in the mountain areas from the state of Rio de Janeiro, as they point out to a good adaptation of both varieties to this production area.

ACKNOWLEDGEMENTS

Authors thank Mrs. Marcelly C. S. Santos for technical assistance. H.R.B. thanks research grants from Conselho Nacional de Desenvolvimento Científico e Tecnológico – CNPq (311021/2021-2) and Fundação Carlos Chagas Filho de Amparo à Pesquisa do Estado do Rio de Janeiro – FAPERJ (210.049/2020). A.F.M. thanks CNPq for a scholarship.

Conflict of Interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

References

- [1] Murakami A., Darby P., Javornik B., et al. (2006). Molecular phylogeny of wild Hops, Humulus lupulus L., Heredity, 97, 66-74.
- [2] Agência Nacional de Vigilância Sanitária, (2019). Farmacopeia Brasileira, 6th ed., ANVISA, Brasília.
- [3] Bizzo H. R., Barboza E. G., Santos M. C. S., Gama, P. E., (2020). A set of electronic sheets for the identification and quantification of constituents of essential oils. *Quimica Nova*, 43, 98-105.
- [4] van den Dool H., Kratz P.D. (1963). A generalization of the retention index system including linear temperature programmed gas—liquid partition chromatography, *Journal of Chromatography*, 11, 463-471.
- [5] Henning J.A., Townsend M.S., Gent, D.H., et al. (2021). Registration of high-yielding aroma hop (*Humulus lupulus* L.) cultivar 'USDA Triumph'. *Journal of Plant Registration*, 15, 244-252.