Some aromatic compounds in honey wine and spirits

Umut Tas[1], Hatice Kalkan Yıldırım[1]

[1] Department of Food Engineering, Faculty of Engineering, Ege University, Izmir, Turkey

IJASR 2022
VOLUME 5
ISSUE 2 MARCH – APRIL **ISSN: 2581-7876**

Abstract: Introduction. Mead is the oldest fermented, traditional alcoholic drink produced by fermentation of diluted honey. The mead spirit is an alcoholic drink made from mead by using distillation. The increasing interest to both drinks lead to new studies. The aim of this study was to determine the main volatile compounds found in mead and mead spirits.

Materials and methods. The procedure for mead production was done by following traditional applications. The distillation process concerning mead spirits was design in laboratory scale units. Volatile compounds in samples were analyzed by Gas chromatograph system equipped with column CP-WAX 57 CB, 0.20 μm x 50 meters, inner diameter is 0.25 mm properties. The calibration standard solutions were prepared by diluted 45:55 (v/v) ethanol-water.

Results and discussion. The highest value of volatile compound was determined in mead as 29.75 g/hL. Concerning the mead spirits the highest value was determined for 3-methyl-1-butanol as 15.41 g/hL. The results demonstrated the importance of used procedures during productions.

Conclusions. These results demonstrated that volatile compounds in mead and mead spirits could be optimized.

Keywords: Honey wine, Honey spirits, Aromatic compounds

Introduction

Honey, a popular sweetener throughout the world, is made by bees generally from nectars extracted from the nectarines of flowers [1]. The composition and flavour of honey varies, depending mainly on the source of the nectar(s) from which it originates and to a lesser locations extent on certain external factors - climatic conditions and beekeeping practices in removing and extracting honey [2].

Mankind has been fermenting foods and beverages for thousands of years, which certainly predates our knowledge that yeasts were the microbial agents responsible for metabolizing sugar into alcohol [3]. Mead is a traditional alcoholic drink derived from the fermentation of diluted honey performed by yeasts and is, perhaps, the oldest fermented drink in the world [4]. Meads contain at least 7% of ethanol and many another compounds such as sugars, acids, vitamins, antioxidants, minerals, etc. [5].

The yeasts used in mead production are starter yeasts, such as strains of *Saccharomyces cerevisiae* and related species, which are more tolerant of ethanol and more competitive for growth in media with high sugar concentrations [6]. Currently, mead makers are looking for ways to introduce aroma and flavor diversity as a means of improving style and increasing product differentiation [7].

In the production of spirits, the initial material for distillation is fermented mash (or juice) that contains ethanol and water as the main compounds and a huge number of other volatile compounds with different boiling points [8].

The compounds with lower boiling points or high solubility in ethanol are distilled earlier. For the same reason, the compounds that are more water-soluble or have higher boiling points are separated later [9].

According to the last beekeeping report of Ministry of Agriculture and Forestry, Turkey is listed as the second country in the world related to honey production [11]. So, there is a great potential for honey wine and honey spirits production. Based on this knowledge considering the geographical effects of honey supplied from Turkey, the study was conducted with purpose to disseminate the results to all countries interested in aromatic compounds of mead and honey spirits

.

Material and Methods

Materials

Used honey was based on pine tree. The honey collected from Aegean region in Turkey (37° 13' N, 28° 21' E). The brix value was determined as 80.4 and the total sugar content was detected as 68.51% (w/w).

The production steps of mead

During fermentation as a culture were used *Saccharomyces cerevisiae* (Oenoferm C2 yeast Erbslöh Geisenheim, Germany). The inoculum of *Saccharomyces cerevisiae* was as 275 g/hL in accordance to the required procedure.

The honey was diluted with water until the brix value of 25.5 and the temperature was adjusted to 22°C during fermentation for 264 hours. The fermenter was filled up to 80%.

The production steps of honey sprit

Distillation is the operation often used in the chemical and food industry to separate a liquid mixture into products based on their different volatilities [12]. The purpose is typically the removal of a light component from a mixture of heavy components, or the other way around, the separation of a heavy product from a mixture of light components [13].

For distillation of our materials, 250 mL sample added into flask. After that the flask was assembled to distillation apparatus (Figure 1). The distillation apparatus used for distillation of mead contain following parts: 1- distilling flask 1 liter, 2- vigorous distilling column 20 cm, 3- west condenser 10 cm, 4- condenser 40 cm and flask heater [14].

During the distillation are collected three fractions: "the head", "the heart" and "the tail". The heads part contains higher concentration of low boiling point components which are mainly undesirable compounds. These compounds gives the distillates an unpleasant, strong and sharp flavor [8]. The best fraction is "the heart" part which contain highest concentration of ethanol. Therefore, the first and last factions were discarded and the second fraction was proceeding as the required and analyzed.

Chemical Analyses

Sugar content analyses

The sugar content was analyzed by Chromatographic technique. The analysis was conducted on a Shimadzu Prominence-i LC-2030C liquid chromatograph with refractive index detector (Japan) and Inertsil® NH2, 5 µm 4.6 x 250 mm (5020-05546 model). Mobile phase was prepared by using acetonitrile and water ratio of 80:20 (v/v). The flow rate was 1.3 mL/min.

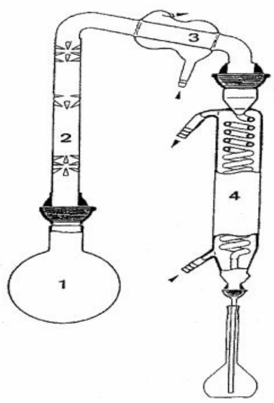

Figure 1. Distillation apparatus

Preparation of standards and sample

Used standards namely fructose, glucose, sucrose and maltose were diluted with water. The dilutions rates for each component were as 1 g/L, 5 g/L, 50 g/L, 100 g/L and 200 g/L. The responses were measured as peak areas vs concentration.

The raw material (1g honey) was diluted by 200 mL water then the sample was kept for 10 min in ultrasonic water bath. The response measured as peak vs concentration.

Volatile Compounds Analyses

Volatile compounds were analyzed using gas chromatograph system. The Shimadzu Nexis GC-2030 gas chromatography system is equipped with HS-10 head space sampler unit (Japan) and CP-Wax 57 CB 50 m x 0.25 mm x 0.20 µm column. The calibration standard solutions were prepared by dilution with 45:55 (v/v) ethanol-water solution ratio (Table 1). All chemicals were with analytical grade (Merck).

Table 1. Standard solutions for GC calibration (mg/100 mL)

Components	Mix-1	Mix-2	Mix-3	Mix-4	Mix-5	Mix-6
Acetaldehyde	1.54	3.86	23.17	38.61	77.22	154.44
Methyl Acetate	1.84	4.60	27.62	46.04	92.07	184.14
Ethyl Acetate	1.78	4.46	26.73	44.55	89.10	178.2
Methanol	1.58	3.94	23.65	39.42	78.84	157.68
2-Butanol	1.60	4.01	24.06	40.10	80.19	160.38
1-Propanol	1.60	3.90	23.95	39.92	79.84	159.68
Iso-butanol	1.59	3.97	23.82	39.70	79.40	158.80
N-Butanol	1.62	4.04	24.25	40.42	80.84	161.68
2-Methyl-Butanol	1.16	4.02	24.11	40.18	80.36	160.72
3-Methyl-Butanol	1.59	3.97	23.81	39.70	79.38	158.76
Furfural	1.14	2.84	17.05	28.42	56.84	113.68

Flow rates of gases and temperature values used during GC analysis

The oven temperature of head space unit was 120°C, transfer line temperature was 180°C and the injection temperature selected as 190°C. Carries gas was N_2 and the pressure was adjusted as 120 kPa. The flowrate was 57.7 mL/min. The column temperature was 40°C for 1 min. The rate of temperature changes were as: 8°C/min to 100°C followed by 20°C/min to 210°C.

Alcohol Analysis of Mead

The alcohol was analyzed by using digital ebulliometer. The quantity of 35 mL pure water was added to the sample chamber then the ebulliometer was calibrated via boiling point of pure water. After washing the chamber with honey mash, 35 mL sample was added to the chamber and alcohol content was determined by using graphical panel of equipment.

Alcohol Analysis of Distillated Mead

After completing the distillation, the amount of 30 mL of distillate was taken and the density was measured by electronic densitometer at temperature of 20°C. For this measurement, Anton Paar DMA 35 v4 electronic densitometer was used.

Result and Discussion

The mead spirit is an alcoholic drink made from mead by using distillation technic. The purpose of this study was to find out the main volatile compounds found in mead and mead spirits.

The fermentation was completed within 264 hours. The initial pH was 4.72 the final pH was determined as 3.67.

The initial values of other parameters such as brix, sugar and alcohol contents were as 25.5°brix, 21.73 g/L, and 0%, respectively. The final values of these parameters were determined as 15°brix, 15.31 g/L, and 10.68%, respectively. (Figure 2).

As was expected yeasts metabolize sugars, such as glucose and fructose to form ethanol and carbon dioxide. Hence, sugar concentration and brix value decrease alcohol concentration increase during fermentation.

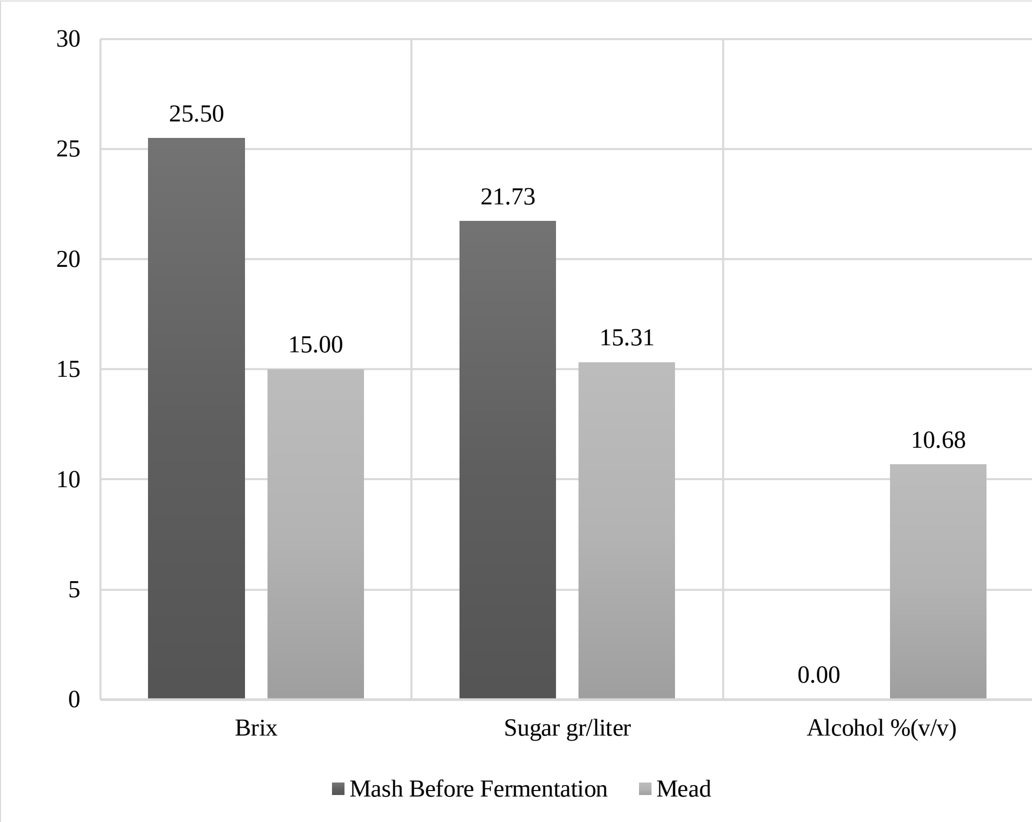

Figure 2. Comparison brix, sugar and alcohol values between unfermented mash and unfermented mead

The volatile fractions of the mead and distillated mead were analyzed and demonstrated in Figure 3. A total of 8 different volatile compounds were identified. These were acetaldehyde, methyl acetate, ethyl acetate, methanol, 1-propanol, isobutanol, 2-methyl-1-butanol, 3-methyl-1-butanol. The boiling points of these compounds are as 20.2°C, 57.1°C, 77.1°C, 64.7°C, 97°C, 108°C, 128.7°C, 131°C, respectively.

Distillation is a method of separating mixtures based on differences in their boiling points. Simple distillation refers to the separation of compounds by evaporating the liquid and collecting it after it passes through a condenser to be changed into a liquid state [15]. The more-volatile components tend to concentrate in the vapor and the less-volatile components in the liquid [16].

The concentration of volatile compounds in distillated mead is significantly different from fermented samples.

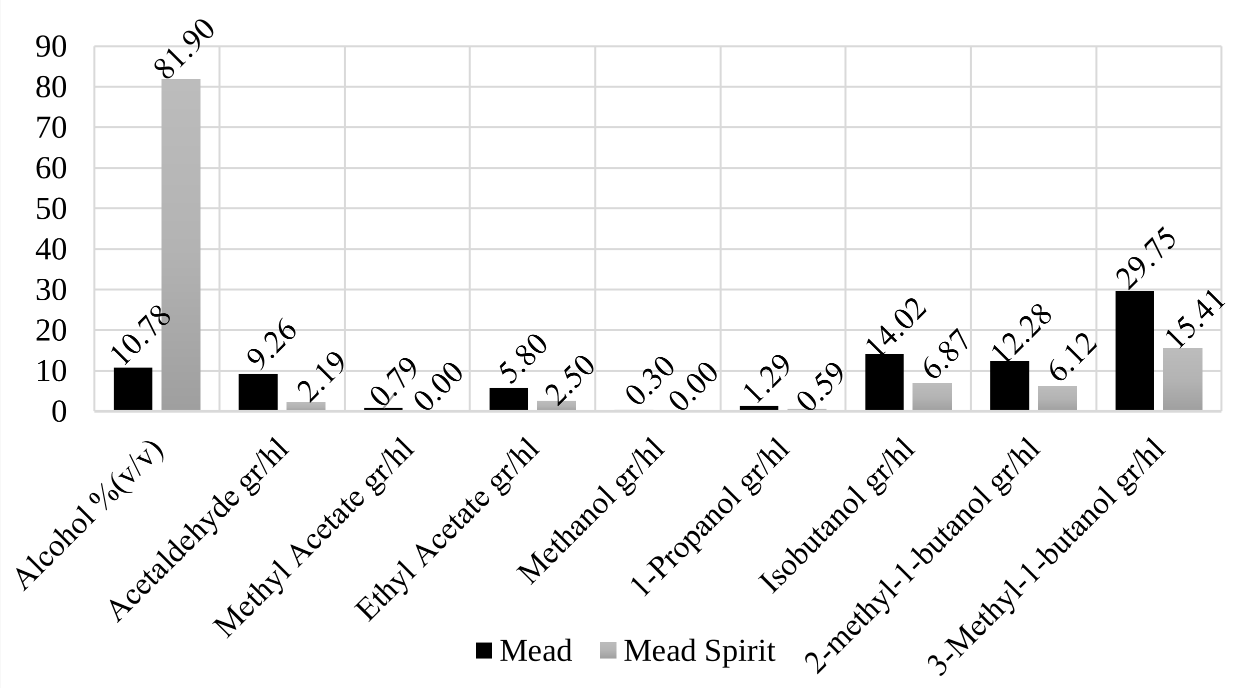

Figure 3. Comparison some parameters mead and honey sprits

During the experiment, 250 mL sample was added into flask distillated. Obtained first fraction of 10 mL distillated volume was discarded. The procedure continued until got 30 mL distillated. The rest amount of remained in the flask to be redistilled.

The more-volatile components as acetaldehyde, methyl acetate, ethyl acetate and methanol tent to concentrate in the vapor and discarded within first fraction (10 mL). The compounds as 1-propanol, iso-butonol, 2-methyl-1-butonol, 3-methyl-1-butonol as the less-volatile components remained in the flask. Hence, the concentration of distillated mead volatile compounds found less than that of fermented volatile compounds.

Considering comparison of each volatile compound obtained by fermentation and distillation interesting results were obtained.

The ethanol alcohol value (%) for fermented honey was 10.78 for distiled was determined as 81.90. The values of acetaldehyde 9.26/ 2.19; methyl acetate 0.79/ nd; ethyl acetate 5.80/ 2.5; methanol 0.30/ nd; 1-propanol 1.29/ 0.59; isobutonol 14.02/ 6.87; 2-methyl-1-butanol 12.28/6.12; 3-methyl-1butanol 29.75/15.41 were determined for mead and mead sprit, respectively.

As were demonstrated from the results the value of ethyl alcohol increased by 659 % after distillation. The acetaldehyde 76 %; methyl acetate 100 %; ethyl acetate 57 %; methanol 100 %; 1-propanol 54 %; isobutonol 51 %; 2-methyl-1-butanol 50 %; 3-methyl-1butanol 48 % decreased after distillation.

In a study done by A.P. Pereira and etc. were demonstrated that meads obtained by different pitching rates and fermented by the two yeast strains have quantitative differences in aroma profiles [17]. These facts confirming the contribution of both yeast metabolism and inoculum size on the sensory characteristics of meads. In general, the total concentration of volatile compounds increased with increasing pitching rate, except for the lowest pitching rate [17].

Li and Sun [18] used different types of honey to be fermented by yeasts for production of different types of meads, such as fruit-honey wines and sparkling wine, which may have different flavors depending on the flower source of the honey, yeast species and additives used during fermentation. The results demonstrated that six (fruity aroma, floral aroma, honey aroma, fatty aroma , taste quality, overall impression) descriptors of all meads were significantly different [18].

Benes et al. [19] investigated the blossom honey meads fermented by three different *Saccharomyces cerevisiae* var. *bayanus* strains in order to compare the production of volatile organic compounds (VOC). Monitoring of the volatile profile of meads led to conclusions that individual yeast strains produced various metabolites in different concentrations under the same fermentation conditions which significantly influenced the final mead flavor [19].

Mendes-Ferreira et al. [20] stated that the popular wine yeast strain such as S. *cerevisiae* activity could be manipulating by addition of diammonium phosphate (DAP) and/or potassium tartrate and malic acid. As a result of DAP addition, the reduced fermentation length, improved breakdown of sugars and generation of more aroma compounds by the yeast strain was achieved [20].

In other study was emphasized that nitrogen addition could effects concentration of volatile compounds values. QA23 and ICV D47 *Saccharomyces cerevisiae* yeast stains used to production of mead. The production of VOC appears to be enhanced by nitrogen, since higher amounts were found in the fermentation supplemented with DAP for both strains QA23 and ICV D47 yeast stains [21].

Honeys were fermented and distillated to compare the volatile organic compounds which are responsible for the secondary aroma of alcoholic beverages.

CONCLUSION

The comparison of mead and mead sprit using distillation demonstrated that the volatile organic compounds decreased after distillation.
The highest value of volatile compound was determined in mead as 29.75 g/hL. Concerning the mead spirits the highest value was determined for 3-methyl-1-butanol as 15.41 g/hL. The results emphasized the importance of used procedures during productions.

REFERENCES

1. Adebiyi F.M., Akpan I., Obiajunwa E.I., & Olaniyi H.B. (2004). Chemical/physical characterization of Nigerian honey. *Pakistan journal of Nutrition*, 3(5), pp. 278-281.
2. White J.W.J. (1975). *Composition of Honey*. Honey, A Comprehensive Survey. E. Crane, Pp. 157–206
3. Barry J.P., Metz M.S., Hughey J., Quirk A., & Bochman M.L. (2018). Two novel strains of *Torulaspora delbrueckii* isolated from the honey bee microbiome and their use in honey fermentation. *Fermentation*, 4(2), pp. 22.
4. Pascoal A., Oliveira J.M., Pereira A.P., Féas X., Anjos O., & Estevinho L.M. (2017). Influence of fining agents on the sensorial characteristics and volatile composition of mead. *Journal of the Institute of Brewing*, 123(4), pp. 562-571.
5. Švecová B., Bordovská M., Kalvachová D., & Hájek T. (2015). Analysis of Czech meads: Sugar content, organic acids content and selected phenolic compounds content. *Journal of Food Composition and Analysis*, 38, pp. 80-88.
6. Querol A., Barrio E.L.A.D.I.O., & Ramón D. (1992). A comparative study of different methods of yeast strain characterization. *Systematic and Applied Microbiology*, 15(3), pp. 439-446.
7. Chen C.H., Wu Y.L., Lo D., & Wu M.C. (2013). Physicochemical property changes during the fermentation of longan (*Dimocarpus longan*) mead and its aroma composition using multiple yeast inoculations. *Journal of the Institute of Brewing*, 119(4), pp. 303-308.
8. Spaho N. (2017). Distillation techniques in the fruit spirits production. *Distillation-Innovative Applications and Modeling*, pp. 129-152.
9. Curvers J.M.P.M., Rijks J., Cramers C.A.M.G., Knauss K., & Larson P. (1985). Temperature programmed retention indices: Calculation from isothermal data. Part 1: Theory. *Journal of High Resolution Chromatography*, 8(9), pp. 607-610.
10. Liu Q. (2014). Analysis of volatile compounds and their changes during liquor aging of chinese liquor 'gujing gongjiu'.
11. (2018), *Beekeeping Statistics*, available at:

https://arastirma.tarimorman.gov.tr/aricilik/Belgeler/istatistik/2018%20YILI%20D%C3%9CNYA%20ARICILIK%20VER%C4%B0LER%C4%B0%2002.03.2020.pdf.
12. de Almeida Viseu M.N. (2014). Dynamic modelling of batch distillation colomns. Master of science thesis, pp. 1-112.
13. Sutter D. and de Albuquerque I.V. (2013). *Practica in Process Engineering II*, IPE Sep. Process Lab. ETH Zurich, no. Phase 1, pp. 30.
14. Anonymous (2017). Türk gıda kodeksi distile alkollü içkiler analiz metotları tebliği, (TEBLİĞ NO: 2017/9).
15. (2019), *Simple Distillation | Distillation | Separation Processes*, available at: https://tr.scribd.com/document/62212912/Simple-Distillation.
16. Zhang W., Taylor P.H., & Darton R.C. (2017). Simple estimation of effective elastic constants for thin plates with regular perforations and an application to the vibration of distillation column trays. *The Journal of Strain Analysis for Engineering Design*, 52(1), pp. 57-66.
17. Pereira A.P., Mendes-Ferreira, A., Oliveira, J. M., Estevinho, L. M., & Mendes-Faia, A. (2013). High-cell-density fermentation of *Saccharomyces cerevisiae* for the optimisation of mead production. Food microbiology, 33(1), pp. 114-123.
18. Li R., & Sun Y. (2019). Effects of honey variety and non-*Saccharomyces cerevisiae* on the flavor volatiles of mead. *Journal of the American Society of Brewing Chemists*, 77(1), pp. 40-53.
19. Benes I., Furdikova K., & Šmogrovičová D. (2015). Influence of *Saccharomyces cerevisiae* strain on the profile of volatile organic compounds of blossom honey mead. *Czech Journal of Food Sciences*, 33(4), pp. 334-339.
20. Mendes-Ferreira A., Cosme F., Barbosa C., Falco V., Inês A., & Mendes-Faia A. (2010). Optimization of honey-must preparation and alcoholic fermentation by *Saccharomyces cerevisiae* for mead production. *International journal of food microbiology*, 144(1), pp. 193-198.
21. Pereira A.P., Mendes-Ferreira A., Oliveira J.M., Estevinho L.M., & Mendes-Faia A. (2015). Mead production: effect of nitrogen supplementation on growth, fermentation profile and aroma formation by yeasts in mead fermentation. *Journal of the Institute of Brewing*, 121(1), pp. 122-128.