Determination of home-made wine selected parameters and study of honey addition impact on pro-healthy components content

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Abstract
The chemical characteristic of home-made wine, based on the vinification process of Maréchal Foch grapes with minimal intervention, was discussed. The addition of honey in the vinification process has been studied to improve the parameters of the pro-health properties of wine. Assays of antioxidant capacity, pH, colour intensity, total acidity, histamine, tyramine, tryptamine and phenylethylamine contents were carried out during the fermentation, maturation and storage processes. Moreover, the discussed above and selected oenological and quality parameters (organic acids, metals, total and free sulphur dioxide, alcohol content) of obtained wines were compared with commercial wines. Obtained results of allergenic compounds (SO₂ and histamine) showed a significantly lower level for home-made wines. The presence of honey during the fermentation process significantly improved the antioxidant parameters, titratable acidity, and influenced the final product colour intensity and colour brilliance. The obtained data show that home-produced wine seems to be a valuable alternative to traditional commercial production due to the lack of chemical additives and potentially allergenic substances in the presence of compounds that enhance human health. Moreover, minimal intervention during fermentation, no filtering step, no chemical additives, processing aids and clarifying substances suggest that home-made wine can be considered natural.

Keywords Home-made wine · Maréchal Foch grape · Vinification process · Antioxidant capacity · Biogenic amines · Oenological and quality parameters

Introduction
Nowadays, the market offers organic wines, biodynamic wines, natural wines, wine without sulphites, or wines with low environmental impact, which results from the growing interest of consumers in these products. Natural wine refers to wine-making free of added plant protection chemicals and other additives. Such wine requires the usage of organic farming, minimal intervention during fermentation, and low histamine and sulphite concentrations in the final product [1]. Home-made alcohol, both from grapes and other fruits, can be considered as natural wine. In most cases, home-made wine is produced without sulphates, chemical fermentation additives, pesticides, herbicides, fungicides, and fertilizers. Moreover, home-made wine is a living product rich in bacteria and compounds that enhance the human microbiome. The latter means that home-made wines with moderate consumption can have a positive effect on human health. Observed increasing consumer interest in wine with high safety and healthy values, home-produced wine seems to be a valuable alternative to traditional commercial production.

While Poland may not seem to be a typical wine country, the home-making traditions in Poland have been cultivated for ages and are still developing. Global warming, new grapevine varieties composed of hybrid types better suited to the Polish climate and cultural changes resulting from the accession to the EU cause dynamic development of viticulture [2]. Additionally, the grapevine cultivars adapted to the harsh climatic conditions give the Polish grape wines some unique sensory features.

In the present study, the selected factors that affect the quality of wine during the home-made wine-making process were determined and discussed. The wine-making process was carried out traditionally and included simple ingredients.
(Maréchal Foch grapes, water, sugar, yeast) without chemical additives. Moreover, to improve the health-promoting parameters of the obtained wine, replacement of half of the added sugar with honey was proposed. Assay of antioxidant capacity (AC), total polyphenol content (TPC), pH, colour intensity (CI), total acidity (TTA), and selected biogenic amine (BA) contents were carried out during the brewing process.

Additionally, oenological and quality parameters, including organic acids (citric, malic, tartaric and succinic), metals (Zn, Cu, Fe, Mn, Ni, Cr, Pb and Mg), total and free sulphur dioxide and alcohol contents in final products were determined. The obtained results were discussed regarding applied wine-making process compounds and compared with commercially available red wine. To my knowledge, no report has been performed to evaluate the quality of homemade wine during the wine-making process and the impact of honey addition on the quality of this process and the final product.

Materials and methods

Analytical grade: 2,9-dimethyl-1,10-phenanthroline (neocuproine), 3,4,5-trihydroxybenzoic acid (gallic acid), 3,5-bis(trifluoromethyl)phenyl isothiocyanate (BPI), tyramine (Tyr), histamine (His), tryptamine (Trp), 2-phe-nyl ethylamine, 99% (Phen), methanol (HPLC grade), acetanilide (HPLC grade), tetrahydrofuran (THF, HPLC grade), hexane (95%), diethyl ether (≥ 99.9%), ethyl acetate (99.8%), 2,4,6-tris(2-pyridyl)-s-triazine (TPTZ, 99%), Trolox (TE, 6-hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid), hydroxyethylcellulose (HEC), 1,3-bis[tris(hydroxymethyl)methylamino]propane (BTP), β-alanine, 2-[(2-aminoacetyl)amino]acetic acid (Gly–Gly) were purchased from Sigma Aldrich (Poland), and sodium hydroxide, sodium chloride, magnesium sulphate (anhydrous), nitric acid (65% ultrapure), sulphuric acid (98%), hydrochloric acid (37%), hydrogen peroxide (30%), Folin and Ciocalteu phenol reagent, copper(II) chloride, iron(III) chloride hexahydrate, ammonium acetate, sodium acetate, acetic acid, sodium bicarbonate (anhydrous), ethanol (96%), citric acid, tartaric acid, malic acid, benzoic acid and succinic acid from Merck (Poland). Single-element standard of Zn, Mg, Fe, Pb, Ni, Mn, Cr and Cu for Atomic Absorption Spectroscopy (standard reference materials from NIST) were purchased from Merck. Deionised water was used for all solutions.

The laboratory centrifuge (MPW-350, Poland, max speed 9000 rpm, RFC 8693×g, angle 30°, falcon tubes 50 mL) was used for sample centrifugation. Deionised water was obtained from a demineralised water supplier (HLP Smart 2000) HydroLab, Poland. Microwave mineralisation of wine samples was performed by a microwave digestion system (ERTEC MAGNUM II, Ertec-Poland) working in the closed system. UV–Vis spectra were recorded using a UNICAM Helios i, a double-beam spectrophotometer with a 1 cm quartz cell. The pH measurements were made with a multifunctional computer meter (Elmetron, Poland). The volatile acidity of wine samples was determined by steam distillation with titration (Vapodest 20, Gerhardt, Germany). The selected metals in wine samples were determined by an atomic absorption spectrometer (iCE 3000 FL Thermo Scientific, United Kingdom). The HPLC system (SHIMADZU, Japan), equipped with an autosampler SIL-20AC HT and a photodiode multi-wavelength detector (SPD-M20A Promi- nence Diode Array Detector), was applied. The chromatographic data were recorded and processed by the LC solution program version 1.23 SP. Analyses were carried out on a Kinetex EVO C18 (5 μm particle size, 150×4.6 mm) at 25 °C. The Villa Labeco EA 102 isotachophoretic analyser (Villa Labeco, Spišská Nová Ves, Slovakia) were applied for the determination of organic acids.

The brewing process

Maréchal Foch (hybrid French) grapes used in the production of wine were coming from grapevines located in north-central Poland. They are grown without any artificial plant protection products or fertilizers. The grapes were harvested manually in half of October, carefully selected by visual inspection to avoid visible yellow dust and mould contaminations, and destemmed. Potassium metabisulphite solution (3%) was used to disinfect wine-making equipment. The linden honey was originated from the producer from the clear region of the northeast of Poland. Fresh, liquid linden honey was clarified at 35 °C.

Preparation of mother yeast

Mother yeast was prepared three days before the grape pressing. The juice (250 mL) was squeezed from the grated apples, diluted 1/1 v/v with boiled water and brought to the boil. The juice cooled down to room temperature was poured into a vessel, a spoonful of sugar and liquid Bordeaux yeast (commercially available) was added and left for 3 days at 25–27 °C without light.

Preparation of must for fermentation

The destemmed and manually crushed grapes were divided into two batches. One batch treated in a standard way: 3 kg of grapes were transferred into the demijohn, and 0.5 kg of sugar dissolved in 1 L of water was added (22 Blg). Next, about 250 mL of mother yeast and 1 g of yeast nutrient (commercially available) were added and mixed up. The demijohn was closed with cotton wool and left warm (20–22 °C). The
second batch was treated analogously, including 150 g of sugar and 350 g of honey.

Must fermentation

Initial fermentation has lasted about 3 days. Both demijohns were gently moved during this stage to distribute the yeast better evenly and facilitate oxygen and nutrients. The turbulent fermentation was ended after 7 days. Next, the second portion of sugar was added (250 g of sugar with 250 mL of water and 200 g of sugar + 300 g of honey + 250 mL of water to the first and the second batch, respectively), and both demijohns were closed with a stopper with a glass airlock and allowed to after-fermentation.

Clarification and maturing of wine

After 8 weeks, the young wine was decanted from above the sediments and the next portion of sugar was added (250 g of sugar with 250 mL of water and 200 g of sugar + 300 g of honey + 250 mL of water to first and second batch, respectively). Next, demijohns were covered with a stopper with a glass airlock and placed in a dark place (temperature about 21 °C). After 5 weeks, wine was decanted again and left for the next four weeks. The clear wine was left to mature for 4 months and then bottled. The wine was stored for a year at a low temperature and protected from light.

Samples and sample preparation

Samples of wine were taken after the following stages of wine production: turbulent fermentation (sample 1 and 2), silent fermentation (sample 1A and 2A), maturation by 4 months (sample 1B and 2B) and 1 year of storage in bottles (sample 1C and 2C). For comparison, four samples of wine created commercially were detected. Two samples were obtained from a small polish vineyard and included organic, red, dry wines created from French varieties: Maréchal Foch 100% (alc: 12%; sample 3) French varieties: Maréchal Foch and Leon Millot (alc: 12%, sample 4). Additionally, samples of two commercially available red wines: sweet dessert Bulgarian wine (alc: 17%, sample 5) and Spanish medium-dry Cabernet Sauvignon (alc: 12%, sample 6).

The obtained wine was slightly cloudy, and before all analysis was centrifuged (9000 rpm) and filtrated. For CUPRAC, FRAP, TPC and ITP methods, wine samples were diluted (1 mL of sample was made up to 50 mL by the deionised water). For selected BA determination procedure of sample preparation was identical as described earlier [3, 4] and included extraction twice with a mixture of hexane, diethyl ether and ethyl acetate (2/1/40 v/v/v). Extracts were dried with anhydrous magnesium sulphate and filtered. Solvents were removed on a rotary evaporator, and the crude products were dissolved in 3 mL of tetrahydrofuran (THF). Next, 100 μL of 3,5-bis-(trifluoromethyl)phenyl isothiocyanate (BPI) in THF was added, and the mixture was stirred at room temperature for 24 h. The solvent was removed, and the product was dissolved in methanol (2 mL), filtered and analysed by HPLC–DAD. For the determination of selected metals, microwave mineralisation procedure was applied. 2 mL of wine was poured with 5 mL of HNO₃ (65%) into the reaction vessel and digested in a closed microwave system. The obtained clear solution was transferred into 50 mL volumetric flasks and made up to the mark with deionised water.

Determination of total polyphenol content (TPC)

The total polyphenol content was measured according to Folin–Ciochette method. Gallic acid was used to obtain a calibration curve with standard solutions within the range of 0.50–4.80 mg/L. The results were expressed as mg/L gallic acid equivalent (mg GAE/L). Correlation coefficient of the calibration curve was 0.9991, whereas detection limit, DL = 0.29 mg/L and quantification limit, QL = 0.97 mg/L.

Antioxidant activity by cupric-reducing antioxidant capacity (CUPRAC) assay

The determination of the total reductive capacity of wine was measured by the CUPRAC method. 2 mL of CuCl₂ solution (0.01 M), 2 mL of neocuproine (0.0075 M) and 2 mL of ammonium acetate buffer (pH 7) were mixed with the appropriate amount of diluted wine samples (0.1–2) and filled up to 10 mL with redistilled water. The mixture was stored for 30 min at room temperature, and the absorbance of the solution is measured at 450 nm. Standard solutions of Trolox, TE (0.05–0.39 mmol/L) were used for the calibration, and the results were expressed as mmol/L of TE equivalents per litre of wine. Obtained results for correlation coefficient, DL and QL were as follows: 0.9994, 0.0094 mmol/L and 0.031 mmol/L, respectively.

Antioxidant activity by ferric-reducing antioxidant power (FRAP) assay

The FRAP reagent, prepared by mixing 25 mL of acetate buffer (pH 3.6), 2.5 mL of TPTZ (10 mmol/L) and 2.5 mL of FeCl₃, was prepared freshly and incubated at 40 °C for 15 min. The appropriate amount of diluted wine samples (0.1–2) and 2 mL of FRAP reagent were transferred into a volumetric flask (10 mL) and made up to the volume with redistilled water. The obtained blue solutions were kept at room temperature for 25 min, and the absorbance was measured at 593 nm. The calibration curve was constructed using eight calibration solutions of TE in the range 2.00–18.00 μmol/L and obtained results for correlation
coefficient, DL and QL were as follows: 0.9993, 0.24 µmol/L and QL = 0.78 µmol/L, respectively.

**Determination of Him, Tyr, Trp and Phen in wine**

The analysis of HPLC–DAD was performed as described previously [4]. The mobile phase was acetonitrile/methanol/water (60:17:23; solvent A) and methanol (solvent B) and the gradient conditions were: 0–5 min A: 100%; 5–7 min A: 99%; 7–12 min A: 92%; 12–15 min A: 80%; 15–17 min A: 85%; 17–18 min A: 90% and 18–25 min A: 100%. The total flow rate was 0.55 mL/min; the injection volume was 20 µL, and the detection wavelength 254 nm. The procedure of derivatization was similar as described by earlier [3]. 400 µL of BPI; 10 mL of THF were mixed at room temperature for 24 h with 0.0550 g of Him, 0.0680 g of Tyr, 0.800 g of Trp and 62 µL Phen. Next, the solvent was removed on a rotary evaporator, and obtained product was dissolved in methanol (50 mL) followed by twofold dilution. Calibration curves of obtained derivatives solutions (calculated as pure amine) were constructed in the range 0.01–0.17 mg/L for Him, 0.01–0.21 mg/L for Tyr, 0.01–0.18 mg/L for Trp and 0.01–0.18 mg/L for Phen. The linearity of curves was in the range 0.9993–0.9998, while DL varied from 0.003 to 0.006 mg/L and QL 0.01 to 0.02 mg/L.

**Determination of organic acids by capillary isotachophoretic method (ITP)**

By ITP method, the following acids: citric acid (CA), tartaric acid (TA), malic acid (MA) and succinic acids (SA), were determined. For analyses of CA, TA and MA the leading electrolyte: 10 mM HCl + 1% hydroxyethylcellulose + 1,3-bis[tris(hydroxymethyl)methylamino]propane + β-alanine to pH = 6 was used, while the terminating electrolyte was 10 mM benzoic acid. Determination of SA was performed with the same terminating electrolyte; whereas the leading electrolyte was 10 mM HCl + 1% hydroxyethylcellulose + 20 mM 2-(2-aminoacetyl)amino]acetic acid, pH 6 was applied as leading electrolyte. Calibration curves of tested acids solutions were constructed in the range: 10.0–100.0 mg/L. The linearity for CA, TA, MA and SA were as follows: 0.9994, 0.9991, 0.9993 and 0.9990, respectively. The levels of DL and QL were below 1.20 mg/L and 3.69 mg/L.

**Determination of selected wine quality parameters**

The colour intensity (CI) of wine samples during the wine-making process was measured as the sum of absorbances at 620, 520 and 420 nm and calculated as a relative percentage of yellow, red, and blue (%Yl, %Rd, %Bl), while colour hue (CH) defined as absorbance ratio at 420 and 520 nm [5, 6]. Additionally, for selected samples (1C, 2C, 3–6) the brilliance of wine was calculated: (100-(1-(A_{620}+A_{520})/2·A_{720})). Titratable total acidity (TTA, g/L of tartaric acid) was determined by titrating with sodium hydroxide solution (0.1 M, endpoint by pH meter). Volatile acidity (VA – g/L of acetic acid) was measured using steam distillation of wine samples (25 mL) followed by titration of NaOH (0.1 M, about 60 °C). The alcohol content was determined based on steam distillation (time: 4 min; steam capacity: 95%) followed by automatic measurement. The total and free content of sulphur dioxide was determined by the titrimetric method. The wine samples (50 mL) were treated with 5 mL of sulphuric acid (25%) and titrated with starch indicator using iodine/iodite titrant (0.01 M) for free sulphur dioxide determination. In the case of total sulphur dioxide, the wine samples (25 mL) were reacted with sodium hydroxide solution (25 mL, 1 M NaOH) for 10 min. Next, 10 mL of sulphuric acid solution (25%), starch indicator and 1 g sodium bicarbonate were added, and the mixture was titrated using iodine/iodite titrant.

**Determination of metals in wine**

Stock standard solutions of Zn, Fe, Cu, Pb, Ni, Mn, Cr and Mg (1000 mg/L) were diluted with 2% of HNO₃ to provide the standard working solutions of Zn, Cu, Pb (10.0 mg/L), Fe, Cr, Mn, Mg (5.00 mg/L) and Ni (1.00 mg/L). Next, solutions for calibration curves were prepared by appropriate dilution of working standard solutions in 2% nitric acid and determined by AAS in the air–acetylene flame. The correlation coefficients of the calibration curves varied from 0.9978 (Ni) to 0.9999 (Mg). Detection limits varied from 0.02 mg/L (Cu) to 1.23 mg/L (Pb), whereas quantification limit from 0.07 mg/L (Cu) to 3.69 mg/L (Pb). In the case of Pb, the observed signal in all samples was too low, and the standards addition method was applied for quantification.

**Statistical analysis**

For each of the tested wines, regardless of the tested parameter, five independent samples were used and analyzed in triplicate. One-way ANOVA was performed to determine the significant differences between data (p < 0.05).

**Results and discussion**

**Wine-making process**

Hybrid grapes (Maréchal Foch) is considered as a variety that provides good quality wine with an attractive, purplish
colour. The red wine-making was based on maceration and alcoholic fermentation, followed by malolactic fermentation. The last phase was the wine aging that including maturation (oxidative aging) and bottling (reductive aging). The wine was naturally clarified and unfiltered. All activities performed manually. Moreover, the addition of honey in the vinification process has been proposed to improve the pro-health properties of wine. The final product of the vinification process was unclarified and slightly cloudy wine without the chemical additives.

The results of TPC, AC, pH, CI, TTA and BAs contents during the turbulent and silent fermentations, oxidative and reductive stages are listed in Table 1.

Initially, the must used for starting the fermentation was analysed for TPC, CUPRAC and FRAP, and results were as follows: TPC: 331.00 ± 4.12 mg/L, CUPRAC: 3.97 ± 0.05 mmol/L, and FRAP: 2.55 ± 0.04 mmol/L, whereas for must with honey the respective results were: 478.80 ± 1.88 mg/L; 4.85 ± 0.04 mmol/L and 3.02 ± 0.04 mmol/L, respectively. The rapid increase of TPC value was observed during alcoholic fermentation, followed by malolactic fermentation. The same trend was noticeable for the CUPRAC and FRAP determination. It is related to the extraction of phenolic compounds, which starts after crushing the grapes, and it is facilitated with the ethanol raising level during the fermentation process [7]. The analyzed parameters decreased during the oxidative and reductive aging of wine. Similar trends were described by Wojdylo et al. [8].

Changes in polyphenolic composition occur due to numerous reactions during wine-making and aging and have an essential contribution to wine sensory properties colour, taste, aroma, mouth feel and astringency. The highest results for all samples were noted for TPC assay due to its highest redox potential in the alkaline medium. However, the Folin test measures all phenolic species and antioxidants and is a non-selective assay but is still prevalent in wine analysis [9, 10]. Comparing the results for proposed wine-making ways, it is evident that the presence of honey significantly improves the antioxidant parameters of wine. After 1 year in the bottle, the differences in the TPC, CUPRAC and FRAP data for the discussed trials were as follows: 379.60 mg/L, 4.39 mmol/L and 3.46 mmol/L. The transfer of phenolic compounds from grapes to the must during vinification is closely related to the type of grapes, growing conditions and the wine-making technique [8, 9]. In this experiment,

| Parameter | Sample 1 | Sample 2 | Sample 1A | Sample 2A | Sample 1B | Sample 2B | Sample 1C | Sample 2C |
|-----------|----------|----------|-----------|-----------|-----------|-----------|-----------|-----------|
| TPC (mg/L) | 909.1±3.98 | 1213.7±4.36 | 1035.8±6.90 | 1370.4±6.14 | 920.9±2.63 | 1304.0±4.53 | 915.7±3.45 | 1295.6±7.25 |
| CUPRAC (mmol/L) | 7.91±0.05 | 11.2±0.03 | 8.78±0.07 | 13.9±0.03 | 7.58±0.04 | 12.6±0.03 | 6.51±0.03 | 10.9±0.03 |
| FRAP (mmol/L) | 5.51±0.03 | 9.84±0.04 | 5.96±0.05 | 10.1±0.05 | 5.18±0.02 | 9.30±0.02 | 5.08±0.03 | 8.54±0.034 |
| CI | 2.21±0.01 | 2.30±0.02 | 2.12±0.02 | 2.07±0.01 | 1.85±0.08 | 1.76±0.02 | 1.50±0.02 | 1.43±0.01 |
| CH | 1.50±0.03 | 1.05±0.02 | 2.11±0.04 | 1.09±0.02 | 1.44±0.05 | 1.24±0.03 | 0.76±0.03 | 0.75±0.01 |
| Yellow (%) | 57.0±0.63 | 48.8±0.50 | 61.8±0.35 | 48.7±0.55 | 51.1±1.07 | 49.2±0.60 | 37.2±1.15 | 36.9±0.17 |
| Red (%) | 37.9±0.44 | 46.7±0.45 | 29.4±0.47 | 44.6±0.44 | 35.4±0.58 | 39.5±0.48 | 48.9±0.84 | 49.0±0.20 |
| Blue (%) | 5.10±0.24 | 4.50±0.09 | 8.77±0.15 | 6.70±0.14 | 13.5±1.22 | 11.2±0.13 | 13.9±0.11 | 14.1±0.05 |
| pH | 3.47±0.02 | 3.38±0.02 | 3.55±0.01 | 3.56±0.02 | 3.52±0.03 | 3.50±0.02 | 3.58±0.02 | 3.60±0.02 |
| TTA (g/L) | 21.4±0.02 | 10.9±0.02 | 7.95±0.02 | 9.36±0.02 | 7.51±0.02 | 7.91±0.03 | 7.21±0.02 | 7.52±0.03 |
| Him (mg/L) | 0.59±0.01 | 0.66±0.03 | 1.30±0.02 | 1.41±0.02 | 1.40±0.03 | 1.43±0.02 | 1.41±0.02 | 1.43±0.01 |
| Trp (mg/L) | 0.74±0.04 | 0.88±0.02 | 1.56±0.03 | 1.65±0.02 | 1.60±0.01 | 1.65±0.02 | 1.62±0.02 | 1.69±0.02 |
| Phen (mg/L) | nd | nd | 0.67±0.03 | 0.71±0.02 | 0.72±0.02 | 0.74±0.02 | 0.74±0.03 | 0.74±0.03 |

Sample 1—wine after turbulent fermentation; sample 1A—wine after silent fermentation; sample 1B—wine after maturation by 4 months; sample 1C—wine after 1 year of storage in bottles; sample 2—wine with honey after turbulent fermentation; sample 2A—wine with honey after silent fermentation; sample 2B—wine with honey after maturation by 4 months; sample 2C—wine with honey after 1 year of storage in bottles

Different letters (a–h) within the same row indicate significant differences (one-way ANOVA and Duncan test, p < 0.05); sorted from the lowest to highest values, where “a” was the lowest

n number of independent sample (each sample analysed in triplicate), TPC total polyphenol content, CUPRAC cupric-reducing antioxidant capacity, FRAP ferric-reducing antioxidant power, TTA titratable acidity, CI colour intensity (420 nm + 520 nm + 620 nm), CH colour hue (420 nm/520 nm), Him histamine, Tyr tyramine, Trp tryptamine, Phen phenylethylamine

A Gallic acid equivalent (mg GAE/L)

B Trolox equivalent

C Expressed as tartaric acid
traditional maceration involves the storage of crushed grapes for several days at low temperature. The results of TPC, CUPRAC and FRAP analyses suggest satisfactory antioxidant capacity parameters for wine with honey.

The modification of polyphenolic contents during wine-making and bottle aging were related to changes in the wine colour (Table 1). The value of CI for both samples was on a similar level at the end of alcoholic fermentation (AF). The systematic decrease was noted during wine-making process and finally reached an equivalent level. The hue (CH) fluctuations were observed for both samples, whereas the final values for both wine samples were the same and comparable with those discussed for young wines [11]. Generally, the higher proportion of the yellow tones to the red one affected the CH value of wine [6]. In this study, the dominant participation of the yellow colour was observed during AF, MLF and maturation (Table 1). However, after storage, the percentage of pigments indicated a significant red colour concerning yellow and blue. In the case of the latter systematic increase was noted. The unique chemistry of hybrid grapes such as Maréchal Foch causes higher colour intensities in wines, often characterized by bluish tones [12]. In hybrid red wines, the dominance of diglucoside anthocyanins, and low concentrations of extractable condensed tannins compared to other grape species affects the wine colour [13]. Wines after 1 year of storage were characterized by similar CI, CH and the percentage of pigments, despite initially different CH values. Examined CH and percentage of red, yellow and blue colour differences were not statistically significant (p ≤ 0.05).

Titratable acidity (TTA) and pH are other essential parameters of wine quality. Both increase the microbiological and physicochemical stability of wine and affect the taste of wine. The lowest pH value for tested wines was observed after turbulent fermentation and increased during the vinification stages. After a year of storage, the pH value has increased to 3.58 (sample 1C) and 3.60 (sample 2C). It is evident that despite the large discrepancy in the pH value at the beginning of the wine-making, these data in the final samples were similar. In the case of TTA (expressed as tartaric acid), the observed data decreased systematically, and after 1 year, 7.21 g/L and 7.52 g/L were noted. The higher TTA for wines with honey was observed during all processes, and the most significant difference in values (1.40 g/L) was detected after MLF. Manns et al. [13] discussed the impact of selected parameters during vinification from the hybrid red grape, such as Maréchal Foch, and TTA values were 11.61 g/L for must before fermentation and 8.10 g/L in wine.

The content of BAs is an important parameter tested during wine-making. Among the many presents in the wine, the aromatic and heterocyclic amines (Him, Tyr, Trp and Phen) have toxicological effects and can affect human health and lead to low-quality wines [14]. Phen has not been detected in studied wine samples regardless of added compounds (sugar or honey). Trp has been found in samples after MLF, whereas Him and Tyr were present from the beginning of wine-making. Moreover, MLF process resulted in a significant increase in BAs content. According to Constantini et al. [15], BAs in wines are mainly formed during malolactic fermentation. This stage is usually considered as one of the most crucial factors for BAs production. It should be noted that honey, as a potential source of BAs, did not significantly affect the final content of these compounds in wines.

There are no references on these compounds determination in home-made wine from Maréchal Foch grapes. However, the contents of BAs in home-made wine from different fruits was discussed by Płotka-Wasylka et al. [16], and obtained values were as follows: not detected (nd)—1.456 mg/L (Him); nd—0.052 mg/L (Trp); nd—3.78 mg/L (Tyr) and nd—0.071 mg/L (Phen).

Additionally, the selected quality parameters: total and free SO₂, alcohol content, volatile acidity, tartaric (TA), citric (CA), malic (MA), and succinic (SA) acids levels and selected metals level of the obtained home-made wine was examined and obtained results are listed in Table 2.

Average concentrations of alcoholic strength were different between tested home-made wines. The higher alcohol content in wine without honey suggests a more efficient fermentation process. On the other hand, the volatile acidity for both wine samples was identical and low, which indicated a lack or low level of microbial spoilage.

No sulphur was added during the vinification process; however, not significant amounts of sulphur compounds were formed during the fermentation process. For this reason, the content of total and free SO₂ was examined in home-made wine samples. The level of total SO₂ in both samples varied between 11 and 13 mg/L, whereas free SO₂ was below the limit of detection.

The content of organic acids in the tested wines decreased as follows: TA, MA, SA and CA. These acids in wine contribute to their organoleptic (physical stability, quality and sensory perception) and nutritional properties [17, 18]. In general, TA, MA and CA are the primary acids in wine grapes, and these acids also contribute the highest proportion of titratable acidity of the wine. On the contrary, several other organic acids, such as SA, lactic and acetic acid, are produced, out of which succinic acid is the most important during ALF. The chemical characteristic of a wine made from interspecific hybrids grown in Poland was discussed by Kapusta et al. [2], and obtained contents of CA, TA, MA and SA were as follows: 0.03 g/L; 1.28 g/L; 0.68 g/L and 0.57 g/L for Maréchal Foch wine, and 0.14 g/L; 1.42 g/L; 0.78 g/L; 0.79 g/L for Leon Millot wine.

Metals affect the organoleptic characteristics of wine, including flavour, freshness, aroma, colour and taste during
On the other hand, some of them are toxic in excessive intake and specific rules restricting the metal content of wine in many countries [17, 19]. From the toxic metals considered for analysis, Zn, Cu and Pb were determined. The acceptable level of these metals in home-made wines has not been defined. However, comparing obtained results (Table 2) with the maximum content suggested by OIV (1 mg/L for Cu; 0.15 mg/L for Pb and 5.00 mg/L for Zn), and Pb recommended in Poland (0.2 mg/L), the tested wines revealed lower level than mentioned. The contents of Pb, Zn and Fe in home-made wines reported by Płotka-Wasylka et al. [20] varied from 24.56 to 29.78 µg/L; 97–132 µg/L and 1231–1456 µg/L, respectively. Comparing these samples with those described by Płotka-Wasylka et al. [20] shows that higher levels of these metals were determined. The content of metals in wine can be attributed to the natural sources and the contamination during the wine-making process. In this study, the environment of grape growth and oenological procedure can be considered as sources of these elements. The higher content of tested metals was determined in wine produced with the honey addition. The calculated differences varied from 0.001 mg/L for Pb to 7.60 mg/L for Mg.

## Comparison to wines commercially available

The same parameters were determined in four wines purchased from the local chain stores for comparison with the studied ones. Two samples were obtained from a small polish vineyard and included organic, red, dry wines (samples 3 and 4), red, sweet dessert Bulgarian wine (sample 5) and Spanish medium-dry Cabernet Sauvignon (sample 6). The obtained results are listed in Tables 3 and 4.

Comparing the results of AC assay for home-made (Table 1) and commercially wine (Table 3), it is evident that all disused parameters were significantly lower for home-made wine or at a similar level (sample 5, Bulgarian red wine). According to Cueva et al. [21], the number of polyphenols in young red wines varies in 900–1400 mg/L. For comparison, total phenol contents in Polish red wine varied from 608.1 to 1860.8 mg/L, and wine made from Maréchal Foch was characterized by 1291.1 mg/L of TPC [2].

The Pearson correlation coefficient (R) was calculated to determine the dependence between discussed tests (TPC, CUPRAC and FRAP). Obtained results correlated well with each other due to the electron transfer based similar mechanism. The values of R were as follows: 0.9580 for TPC-CUPRAC; 0.9451 for TPC-FRAP and 0.9847 for CUPRAC-FRAP (p < 0.001). The highest correlation for CUPRAC-FRAP results comes out from similar redox potentials. However, CUPRAC is sensitive to a wider variety of antioxidant compounds. Similarly, TPC-CUPRAC

### Table 2

| Sample | Alcohol (%) | Volatile acidity | CA (g/L) | TA (g/L) | MA (g/L) | SA (g/L) | Zn (mg/L) | Cu (mg/L) | Ni (mg/L) | Cr (mg/L) | Pb (mg/L) | Fe (mg/L) | Mg (mg/L) | Mn (mg/L) | Fe (µg/L) |
|--------|-------------|------------------|---------|---------|---------|---------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
| 1C     | 12.6b±0.02  | 0.17a±0.005     | 0.10a±0.00 | 2.03a±0.07 | 1.30a±0.01 | 1.18a±0.01 | 1.85a±0.05 | 0.35a±0.02 | nd       | nd       | 0.032a±0.0004 | 2.52a±0.04 | 46.2a±0.04 | 0.92a±0.002 |
| 2C     | 12.1a±0.08  | 0.17a±0.004     | 0.12a±0.01 | 2.01a±0.08 | 1.32a±0.01 | 1.29a±0.01 | 2.19a±0.04 | 0.40b±0.03 | nd       | nd       | 0.033b±0.0003 | 2.94b±0.04 | 53.8b±0.05 | 0.98b±0.003 |

Different letters (a–b) within the same column indicate significant differences (one-way ANOVA and Duncan test, p < 0.05); sorted from the lowest to highest values. Where “a” was the lowest number of independent sample (each sample analysed in triplicate). CA = citric acid, TA = tartaric acid, MA = malic acid, SA = succinic acid.

Expressed as acetic acid.
Table 3  The results of physicochemical, oenological parameters and antioxidant capacity determination in commercially wine (mean value ± standard deviation, \( n = 5 \))

| Sample | TPC (mg/L)\(^a\) | CUPRAC (mmol/L)\(^b\) | FRAP (mmol/L)\(^b\) | CI | CH | Yellow (%) | Red (%) | Rho (%) | pH | TTA (g/L)\(^c\) | CA (g/L) | TA (g/L) | MA (g/L) | SA (g/L) | SO₂T (g/L) | SO₂F (g/L) |
|--------|-----------------|------------------|------------------|---|----|-----------|--------|---------|---|----------------|----------|----------|---------|---------|-----------|-----------|
| 3      | 257.0±4.56      | 12.5±0.01        | 9.56±0.03        | 2.0±0.01 | 45.0±0.49 | 12.5±0.15 | 3.60±0.02 | 6.90±0.02 | 0.22±0.01 | 1.80±0.03 | 1.14±0.03 | 1.07±0.05 | 38.1±0.16 | 7.18±0.03 |
| 4      | 256.3±5.51      | 14.2±0.01        | 11.47±0.05       | 1.49±0.01 | 41.5±0.41 | 48.3±0.29 | 10.2±0.28 | 3.55±0.02 | 6.20±0.02 | 0.29±0.01 | 2.16±0.05 | 0.87±0.04 | 0.89±0.02 | 32.4±0.20 | 6.92±0.13 |
| 5      | 27.3±6.13       | 8.18±0.05        | 7.25±0.05        | 1.81±0.01 | 9.2±0.01  | 38.5±0.25 | 41.8±0.21 | 19.7±0.07 | 3.58±0.03 | 4.35±0.01 | 0.83±0.01 | 0.89±0.02 | 0.65±0.04 | 1.76±0.02 | 56.8±0.14 | 12.1±0.09 |
| 6      | 204.6±7.15      | 12.4±0.06        | 8.69±0.01        | 1.59±0.03 | 0.78±0.03 | 40.1±0.99 | 51.1±0.88 | 8.80±0.16 | 3.46±0.03 | 5.09±0.01 | 0.50±0.01 | 1.85±0.09 | 0.97±0.01 | 0.72±0.02 | 43.3±0.75 | 8.85±0.03 |

Different letters (a–d) within the same column indicate significant differences (one-way ANOVA and Duncan test, \( p < 0.05 \)); sorted from the lowest to highest values, where “a” was the lowest.

\(^a\)Gallic acid equivalent (mg GA/L)
\(^b\)Trolox equivalent
\(^c\)Expressed as tartaric acid

Table 4  The results of biogenic amines and metals determination in commercially wine (mean value ± standard deviation, \( n = 5 \))

| Sample | Him (mg/L) | Tyr (mg/L) | Trp (mg/L) | Phen (mg/L) | Zn (mg/L) | Cu (mg/L) | Ni (mg/L) | Cr (mg/L) | Pb (mg/L) | Fe (mg/L) | Mg (mg/L) | Mn (mg/L) |
|--------|------------|------------|------------|-------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|
| 3      | 2.80±0.07  | 1.90±0.05  | 1.09±0.05  | nd          | 1.80±0.02 | 0.078±0.002 | nd       | 0.15±0.003 | 0.043±0.004 | 2.20±0.02 | 97.6±0.56 | 0.12±0.002 |
| 4      | 3.02±0.05  | 2.00±0.03  | 1.07±0.04  | nd          | 2.18±0.02 | 0.12±0.03  | 0.019±0.0001 | 0.14±0.003 | 0.052±0.0002 | 1.45±0.03 | 102.8±1.02 | 0.11±0.002 |
| 5      | 6.23±0.08  | 6.03±0.09  | 2.12±0.02  | 1.02±0.04   | 3.18±0.04 | 0.92±0.02  | 0.023±0.0002 | 0.28±0.003 | 0.062±0.0001 | 1.81±0.13 | 84.8±0.05 | 0.13±0.003 |
| 6      | 7.02±0.02  | 3.03±0.07  | 1.92±0.05  | 1.67±0.06   | 2.91±0.08 | 0.22±0.02  | 0.022±0.0001 | 0.22±0.002 | 0.058±0.0003 | 1.04±0.06 | 70.5±0.21 | 0.14±0.004 |

Different letters (a–d) within the same column indicate significant differences (one-way ANOVA and Duncan test, \( p < 0.05 \)); sorted from the lowest to highest values, where “a” was the lowest.
correlation corresponds to the CUPRAC mechanism of phenolic hydroxyl group oxidation to quinones.

The CI of commercially available wines varied from 1.46 (sample 6) to 2.05 (sample 4), whereas colour hue from 0.78 (sample 6) to 0.94 (sample 3). The increase of the hue value of red wine can be related to the aging process, which causes a shift from purple-red via brick red to brown tones. According to Skendi et al. [11], CH indicates the development of colour towards orange during aging, with young wines showing values < 1 and aged wines reaching an upper limit of around 1.2–1.3. Obtained values of CH for all commercial samples are typical for young wines. The relative percentage of yellow, red and blue colour indicated that tested wines were dominated by red and yellow with a smaller presence of blue. Additionally, the brilliance calculated for: 1C, 2C, 3–6 samples revealed values: 44.0%, 47.9%; 38.9%; 46.4%; 30.3% and 52.0%, respectively. The obtained data are in good agreement with reported for young wines (40 and 60), whereas lower values for older ones [12].

In samples 3–4, higher TPC contents resulted in higher wine colour intensity (CI, Table 3). However, the discussed relationship was not observed for sample 6, which can be related to the parameters such as raw materials, procedure and storage condition. Moreover, the stabilization of wine colour can be correlated with pH value and acidity. Based on the results in Tables 1 and 3, wines with pH ca 3.6 exhibit bluish tint (samples 1C, 2C, 3 and 5).

When discussing TTA, it can be noted that wines manufactured in a cold climate from hybrid grapes (home-made wine, samples 3 and 4) revealed higher results. The latter can be related to a lower sugar content, which results in a higher content of acids in the must [2]. The high acidity of wines stops the growth of bacteria and other microbes, resulting in a stable product.

The tested samples revealed variations in the content of discussed acids (Table 3). Higher tartaric acid content is evident compared to other acids (except sample 5 — sweet dessert wine). The TA level is highest, and this acid represents about 50% of the total acids in wine, while CA content was the lowest in five tested wines (except sample 5). The CA level in home-made wines (Table 2) is noticeably lower (0.10 g/L and 0.12 g/L) concerning commercial wines. CA is added to adjust acidity and chelate metal ions in commercial production to prevent nutrients from precipitation. Moreover, this compound influences the flavour of wines by promoting the perception of “freshness” while, at the same time, promoting microbial instability and the growth of unwanted microorganisms. The latter can explain the higher CA content in commercial wines.

The observed differences in tested organic acids (Tables 2, 3) may be caused by variations in the composition of musts caused by climate conditions and the fermentation process. In comparison, Ivanova-Petropulos et al. [22] analyzed wines produced from Merlot grape (Macedonia) and reported levels of TA, MA, CA, and SA: 2.44 g/L; 0.33 g/L; 0.33 g/L and 0.77 g/L, respectively. Robles et al. [18] reported TA, MA and SA content in various red wines in the range: 0.6–5.6 g/L; from nd to 1.56 g/L and 0.062–0.83 g/L, respectively.

In wine production, sulphites are considered excellent preservatives that prevent oxidation and microbial spoilage of wine. However, in the gastrointestinal system, they rapidly decompose and release sulphites readily available for absorption. Nowadays, sulphites are considered a poisonous and allergenic substance. These compounds may induce relevant adverse health effects after ingestion in hypersensitive individuals. Average concentrations of total and free sulphur dioxide differ significantly between tested wines. It should be noted, that lower values of both parameters were detected in organic wine (samples 3 and 4), whereas the highest concentrations were found in sweet dessert red wine (sample 5). However, the content of this compound in all analyzed samples was below the accepted by low levels, even for organic wine [23].

The content of biogenic amines was another quality parameter of wine tested in this study (Table 4). All tested BAs were determined in samples 5 and 6, while Phen was not found in samples 3 and 4. The noticeable values of the tested BAs, especially for Him, were detected in samples 5 and 6. Moreover, the level of Him was dominant in the analyzed wines. These compounds can be formed from the amino acid by various microorganisms present in the wine at any production stage, aging or storage [24]. Therefore, the BAs content in wines varies greatly. For comparison, wines produced from the Polish vineyard were tested by Płotka-Wasyłka et al. [20] and results for Him, Tyr, Trp and Phen were as follows: 0.688–0.7146 mg/L; 2.013–2.434 mg/L; nd—0.033 mg/L and nd—0.043 mg/L, respectively. Liu et al. [14] determine the level of bio- genic amines in 57 wine samples from 9 countries and reported levels of BAs within the range: nd—10.67 mg/L (Him); nd—18.76 mg/L (Tyr); 0.33–4.73 mg/L (Trp), and 0.09–19.82 mg/L (Phen). Presently, there are no legally regulated BAs limits for wine in any country. However, different countries have established upper limits of this amine in wine, and obtained value of Him for these samples exceeded the limit required in Germany (2 mg/L) in the Netherlands (3.5 mg/L) and Belgium (6 mg/L) [14, 15].

Comparing home-made wine with commercial, it is evident that higher BAs levels were noted for the latter. The calculated sum of the determined amines for home-made wines was 4.52 mg/L (wine without honey) and 4.63 mg/L (wine with honey), whereas for commercial wines varied from 5.79 mg/L (sample 3) to 15.4 mg/L (sample 5). In conclusion, the home-made wine produced in colder climate...
regions may be competitive to the commercially available wines—regarding BA content.

Finally, the level of Zn, Cu, Fe, Mg, Pb, Mn, Ni and Cr was examined in commercial wines (Table 4). The noticeable differences of tested metals content in wine samples (home-made and commercially available) can be observed. The obtained results for wine samples commercially available suggest a good agreement of this study with the described in the literature [17, 19]. All toxic metals were detected in commercial wine samples but, the levels were below the maximum recommended by OIV.

Comparing home-made wine samples with commercially available (Tables 2, 4), no noticeable difference in the content of Zn was observed. However, the lowest Pb and Mn levels and the undetected levels of Ni and Cr can be considered home-made wine advantages. There are few reports in the literature on the metal content of home-made wines. Notwithstanding, home-made wines exhibit the lowest Mg content, while the highest of Fe and relatively high Cu content. The latter is one of the most frequently occurring heavy metals in wine. However, the excessive amount of Cu can contribute to an enhanced rate of oxidative spoilage. The excessive presence of Cu, Fe and Zn has a definite negative effect on the organoleptic properties of wine.

Conclusion

This study reported characteristics of home-made wines produced in a vinification process, with minimal intervention. The addition of honey in the vinification process has been studied to improve the parameters of the pro-health properties of wine. The unclarified and slightly cloudy wine, without the chemical additives, were obtained after both vinification processes and stored for 1 year. It is noteworthy that some of the noticed differences between both wines diminished during 1 year of storage. The observed differences were statistically not significant (at \( p \leq 0.05 \)) for the following parameters: colour hue, \( \% \) of red, yellow and blue colour, the content of organic acid and volatile acid. On the other hand, the higher content of tested metals with lower alcohol content was determined in wine produced with the honey addition. Notwithstanding, the detected contents of toxic metals were below the acceptable level. The presence of honey significantly improved the antioxidant parameters of the obtained wine and titratable acidity. Moreover, the addition of honey influenced the colour intensity of the final product and its colour brilliance.

Comparing home-made wine with commercial or organic ones, it is evident that the results of AC assay were lower. However, the simple vinification process of home-made wine significantly reduced the number of allergenic compounds (\( \text{SO}_2 \) and histamine). Moreover, minimal intervention during fermentation, no filtering step, no chemical additives, processing aids and clarifying substances suggest that home-made wine can be considered natural. Furthermore, the presence of honey during fermentation increased the antioxidant activity of home-made wine and decreased the strength of alcohol. These factors indicate a positive impact home made wines on health quality and antioxidant activity.

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