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The Effect of Drying Conditions on the Presence of Volatile Compounds in Cranberries

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***Abstract*—**the research was accomplished on fresh in Latvia wild growing cranberries and cranberry cultivars. The aim of the study was to evaluate effect of pretreatment method and drying conditions on the volatile compounds composition in cranberries. Berries pre-treatment methods were: perforation, halving and steam-blanching. The berries before drying in a cabinet drier were pre-treated using all three methods, in microwave vacuum drier – using a steam-blanching and halving. Volatile compounds in cranberries were analysed using GC-MS of extracts obtained by SPME. During present research 21 various volatile compounds were detected in fresh cranberries: the cultivar 'Steven' − 15, 'Bergman' and 'Early black' – 13, 'Ben Lear' and 'Pilgrim' – 11 and wild cranberries – 14 volatile compounds. In dried cranberries 20 volatile compounds were detected. Mathematical data processing allows drawing a conclusion that there exists the significant influence of cranberry cultivar, pre-treatment method and drying condition on volatile compounds in berries and new volatile compound formation.

***Keywords*—**volatile compounds, cranberries, convective drier, microwave-vacuum drier

I. Introduction

CRANBERRY (*Vaccinium macrocarpon*, Ait Ericaceae) is a native plant of Latvia. Berry fruits are rich sources of bioactive compounds, such as phenolics and organic acids, which may hold antimicrobial activities [1]. Cranberries also contain Vitamin C (as evidenced by the presence of citric acid) and phytochemicals. Vitamin C is also an important antioxidant. It is important to recall that the antioxidants β-carotene and Vitamin E protect water soluble substances from oxidising agents; Vitamin C protects water soluble substances the same way. Vitamin C is also involved in the metabolism of several amino acids [2].

Food volatile compounds are directly associated with important sensory traits such as flavor and odor. They are secondary metabolites generated by specific metabolic pathways from carbohydrates, amino acids, and fatty acids.

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In berry fruit, the volatile fraction is made of a number of organic compounds, many of which are chiral compounds [3].

Cranberry aroma is characterised by several aromatic compounds, such as 1- and 2-phenylethanol, 3-phenylpropanol, *(E)*-cinnamyl alcohol, 2-(4-hydroxyphenyl) ethanol, 2-(4-methoxyphenyl ethanol, salicylaldehyde and 4-methoxybenzaldehyde. A tart flavour has been attributed to the levels of benzoic acid, although benzaldehyde, 4-methoxybenzaldehyde, benzoate and benzyl esters might significantly contribute to the overall cranberry aroma [4].

Forty-two compounds comprising over 95% of the aroma complex were identified in cranberry juice of the American cranberry (*Vaccinium macrocarpon* Ait.). These consist of 14aromatic compounds, seven terpenes, nine aliphatic alcohols, six aliphatic aldehydes and six other compounds including the two acids, benzoic and 2-methylbutyric. The aromatic compounds (benzaldehyde, benzyl and benzoate esters) and the terpenes appear to be the major contributors to the aroma of cranberry juice. The remaining 5% of the aroma complex contains over 200 components. Although these compounds occur in very small concentrations, they appear to be important in the overall aroma [5].

Drying is one of the oldest methods of food preservation and it is a difficult food processing operation mainly because undesirable changes in quality [6].

Different drying methods are used for drying of fruits, berries and vegetables. Air-drying is the most common method in the drying of foodstuffs. However, this method leads to serious changes in food such as the loss of the taste, colour and nutritional value of the product, decrease in the density and water absorbance capacity and migration of the solutes from the internal part of the drying material to the surface, due to the long drying period and high temperature. Microwave drying has the specific advantage of rapid and uniform heating due to the penetration of microwaves into the body of the product. Microwave energy is capable of polarizing substances. The electrons in the polarized substance are in motion due to the conversion of electromagnetic energy embedded in the substance into kinetic energy. Electrons bump into each other during this electron movement and their energy is converted to heat energy as a result of friction. Thus, the moisture is removed from the product in the microwave drying more rapidly [7].

Compared with convective atmospheric drying, vacuum drying has some distinctive characteristics such as higher drying rate, lower drying temperature and oxygen deficient processing environment etc., these characteristics may help to improve the quality and nutritive value of the dried products. Presently, vacuum drying has been applied for drying of various food materials, the vacuum drying kinetics of many fruits and vegetables has been investigated and the effect of vacuum drying conditions on the drying process and the qualities of dried products has been evaluated [8].

For the better water evaporation during drying process there are known many vegetable and fruit pre-treatment methods such as: halving or slicing [9], blanching in hot water [10], steam-blanching in order to inactivate enzymes [11] and perforation with needle (of 1 mm diameter) [12].

The aim of the study was to evaluate effect of pretreatment method and drying conditions on the volatile compounds composition in cranberries

II. MATERIALS

The research was accomplished on fresh in Latvia wild growing (*Vaccinium oxycoccus* L.) cranberries and cranberry cultivars (*Vaccinium macrocarpon* Ait.) harvested in Kurzeme region in the first part of October, 2010. The cranberry cultivars were: 'Early Black', 'Ben Lear', 'Steven', 'Bergman' and 'Pilgrim'.

III. METHODS

A. Pretreatment of berries

Three methods were used for pre-treatment of berries: perforation, halving and steam-blanching. The berries before drying in a cabinet drier were pre-treated using all three methods and berries dried in microwave vacuum drier – using two pre-treatment methods – steam-blanching and halving. Part of berries was dried in microwave vacuum drier without any pre-treatment (whole berries).

Perforation of berries was realized manually by a needle (1 mm diameter) about 20 pricks equally on all berry surface; halving was realized manually by knife; steam-blanching was realized using "Tefal VC4003 Vitamin+" (Tefal, China) vessel at temperature +94±1 ºC.

B. Convective drying of berries

For air drying experiments, a cabinet dryer "Memmert" Model 100-800 (Memmert GmbH Co. KG, Germany) was used; drying parameters were as follows: temperature 50 ± 1 °C and air flow velocity 1.2 ± 0.1 m/s. Berries were placed on a perforated sieve (diameter − 0.185 m), with the diameter of the holes – 0.002 m.

C. Microwave-vacuum drying process of berries

For drying experiments in vacuum, a microwave dryer „Musson-1" (OOO Ingredient, Russia) was used. The power of installed magnetrons each of four is 640 W. The necessary amount of microwave energy (magnetron minutes) was calculated. The following drying conditions for processing of cranberries in microwave vacuum drier were selected: the first drying stage at 4 magnetrons – energy of 2100 kJ, the second stage at 3 magnetrons – energy of 2520 kJ, the third stage at 2 magnetrons – 1260 kJ and the fourth stage at 1 magnetron – 756 kJ. Temperature in microwave vacuum drier was 36 ± 2 °C.

D. GC-MS analysis of extracts obtained by SPME

For determination of volatile compounds in cranberries the modified method described by Sabovics [13] was used. Volatile compounds were extracted from berry samples using solid-phase microextraction (SPME) with an 85 µm carboxen / polydimethylsiloxane (CAR / PDMS) coating (Co. LLC. Sigma-Aldrich, Germany). Extraction time was 65 min at 40 °C (pre-incubation without the fibre for 15 min, 40 °C). Volatile compounds from fibre were thermally desorbed in the injector of a gas chromatograph PerkinElmer 500 GC/MS (Inc. PerkinElmer, USA) with a capillary column Elite-Wax ETR (60.00 m x 0.25 mm i.d.; DF 0.25 µm) (Inc. PerkinElmer, USA). In this research gas chromatograph oven temperature program started at 40 °C (7 min), programmed from +40to + 160 °C at 6 °C/min and from 160 to 210 °C at 10 °C/min (15 min); helium was used as the carrier gas at an initial flow rate of 1 ml/min and splitless mode was employed in the cases. Mass spectrometer in Electron impact Ionization (EI+ 70 eV) mode with inlet line temperature at + 250 °C and source temperature + 250 °C was used. Acquisition parameters in full scan mode: scanned m/z 40−400. Compounds were identified by comparison of their mass spectra with mass spectral library Nist98.

E. Mathematical data processing

Data are expressed as mean ± standard deviation; for the mathematical data processing p-value at 0.05 was used to determine the significant differences. A three-way ANOVA analysis using IBM SPSS 20.0 was selected to investigate factor effects (volatile compound, cultivar and drying method) and interactions among them. Experiments were carried out in triplicate.

IV. RESULTS AND DISCUSSION

In food and beverage industry, aroma has become one of the most valuable attributes, not only to ensure the consumer acceptance but also to evaluate the food quality. In this respect, the volatile components of some products have been used to establish the geographical origin as well as to detect fraudulent addition [14].

During present research 21 various volatile compounds were detected in fresh cranberries (Fig. 1).

Headspace-solid phase microextraction was used to characterize the volatile compound present in cranberries. Twenty one volatile compounds were isolated and characterized by GC–MS analysis in fresh (Table 1) and in dried cranberries (Fig. 2 and Fig. 3).

Within the present research it was established, that fresh cranberry cultivar "Steven" contain 15 volatile compounds, the cultivars 'Bergman' and 'Early black' – 13, 'Ben Lear' and 'Pilgrim' – 11 and wild cranberries – 14 volatile compounds. Many common volatile compounds were found in a cranberry cultivars and wield cranberries, as 4-penten-2-ol (fruity), 3-cis-hexenyl formate (melon-like and fruity notes respectively); benzaldehyde (almonds), α-1-terpineol (floral), butyric acid (rancid butter), benzyl alcohol (sweet and fruity).

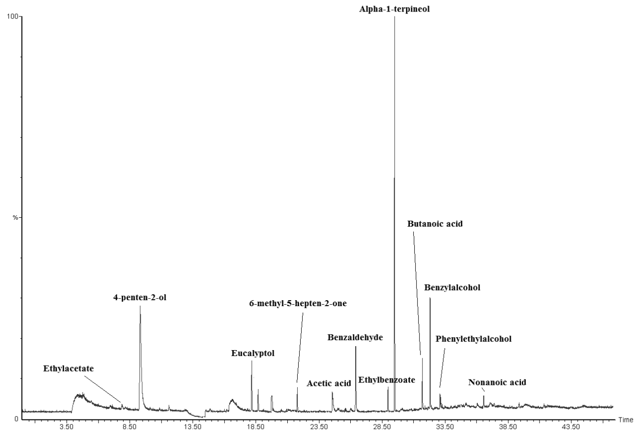

Fig. 1 Chromatogram of volatile compounds in fresh cranberries (the cultivar "Steven")

In scientific literature information was found on volatiles of the cranberries. It is well known, however, that cranberries contain benzoic, quinic, citric, malic acid, from which aroma compounds may, perhaps, be derived. Several aromatic compounds, such as benzaldehyde, anisaldehyde, eugenol and others are known to have high specific odour intensities and also characteristic odour [15].

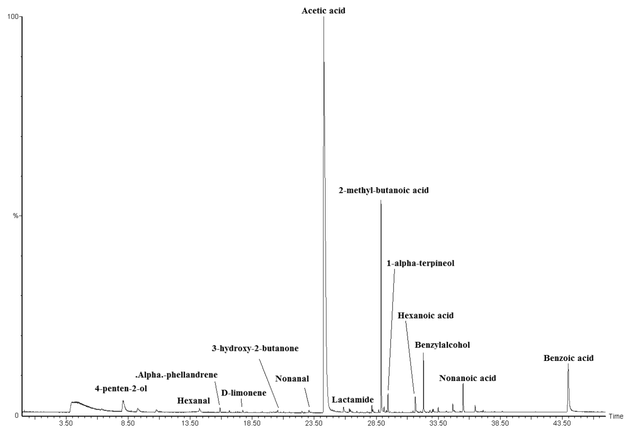

Fig. 2 Chromatogram of volatile compounds in halved microwave-vacuum dried cranberries (the cultivar "Steven")

Benzaldehyde is presented in fresh cranberries in comparatively high amount (Table I). However, the smaller benzaldehyde amount was detected in the cranberry cultivars 'Early Black' and 'Ben Lear'.

In the other scientific source information it was found that the aroma of cranberries is characterised by the presence of several aromatic compounds together with α-terpineol [16].

TABLE I
PEAK AREA (X10^6) OF VOLATILE COMPOUNDS IN FRESH CRANBERRIES

Substance	Cranberry cultivar					
	Steven	Wild	Bergman	Early-black	Benlear	Pilgrim
Ethylacetat	2.31	4.82	4.56	nd	3.98	nd
4-penten-2-ol	31.64	27.36	29.18	13.47	42.03	15.61
Hexanal	nd	1.57	6.14	17.77	7.26	11.25
Eucalyptol	4.13	4.33	1.36	nd	nd	9.63
3-metyl-butan-1-ol	nd	nd	nd	4.66	4.62	nd
(Z)-2-hexanal	1.45	nd	nd	nd	nd	1.88
2-ethyl-3-vinyl oxirane	nd	nd	nd	2.60	nd	nd
Vinylbenzene	nd	6.89	nd	nd	nd	3.63
(Z)-2-heptanal	0.64	nd	nd	nd	nd	nd
6-methyl-5-hepten-2-one	0.85	nd	nd	nd	nd	nd
Hexan-1-ol	nd	1.39	1.50	1.78	2.15	1.30
3-cis-hexenyl formate	1.41	1.21	1.83	2.99	1.60	2.07
Acetic acid	1.69	nd	n.d.	nd	nd	nd
Benz-aldehyde	12.31	10.91	21.87	5.88	7.20	16.84
Ethyl benzoate	1.30	2.82	5.78	0.87	4.18	nd
α-1-terpineol	13.92	9.17	4.78	28.05	4.21	13.55
Butanoic acid	1.90	1.52	2.03	4.34	1.01	1.49
Benzyl alcohol	24.92	26.52	19.68	14.12	21.76	22.74
Phenylethyl alcohol	0.67	1.01	0.67	nd	nd	nd
2-aminooxy propanoic acid	nd	nd	nd	1.50	nd	nd
Nonanoic acid	0.86	0.50	0.61	1.97	nd	nd

*nd – not detected

In the present experiments it was found, that the highest α-terpineol peak area was established for fresh cranberry cultivar 'Steven' ($35.99 \cdot 10^6$), however the lowest − in cranberry cultivar 'Ben Lear' ($1.45 \cdot 10^6$) (Table 1). Therefore it possibly allows predicting more pronounced floral aroma of cranberry cultivar 'Steven'. However, the floral aroma of wild cranberries was not so pronounced (the total peak area was $3.78 \cdot 10^6$). The presence of α-terpineol in berries may be explained by the data found in the scientific literature which revealed that the exocarp and the mesocarp of grape berries contribute directly to monoterpene formation via the methyl-D-erythritol 4-phosphate (MEP) pathway [17], it indicates, and that such formation possibly does not occur in wild berries during growing.

Presence of benzyl alcohol in cranberries is very positive, because benzyl alcohol is a compound with potential antioxidative properties. It has also been shown that the effect of benzyl alcohol – induced aggregation was pH-dependent [18].

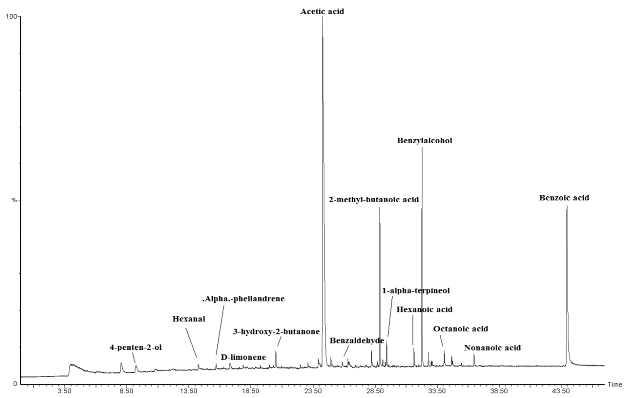

Fig. 3 Chromatogram of volatile compounds in halved convective air dried cranberry (the cultivar "Steven")

The highest peak area of benzyl alcohol in fresh cranberries (Table 1) was detected in wild cranberries ($26.52 \cdot 10^6$), what mainly could be explained with pH value of berries (close to ~3). Therefore high cranberries antioxidative properties can be predicted.

Natural occurring volatiles such as hexanal and (Z)-2-hexanal have a well known antifungal capacity but limited post harvest use due to their volatility. These volatiles have been shown to be a metabolizable fungicide, having adequate volatility and the capability of enhancing the fruit's aroma production by its conversion into other aroma volatiles [19, 20].

The hexanal was not detected in fresh cranberry cultivar 'Steven' (Table 1) and low hexanal presence was found in fresh wild cranberries; therefore the long shelf life could not be appropriable for these berries. However, the (Z)-2-hexanal was not detected in all cranberry cultivars except the cranberry cultivars 'Steven' and 'Pilgrim' (Table 1).

The main component of acidity as acetic acid were detected only in fresh cranberry cultivar 'Steven', what mainly indicate the beginning of oxidation in berries, as a result the beginning of berries spoilage.

Traditionally, benzaldehyde represents the dominant species of aliphatic and aromatic carbonyl compounds, respectively. Benzaldehyde can be considered as a model of simple aromatic aldehydes, and the main role could be as a fumigant [21].

The pronounced presence of benzaldehyde was found in the cranberry cultivar 'Bergman' (peak area was $21.87 \cdot 10^6$).

Drying with moisture reduction provides extended shelf life of fresh fruits and vegetables. Drying methods have different effect on the microstructure and quality of dehydrated products. Drying in conventional dryers at higher temperature usually results in overall quality loss due to surface drying and is also energy intensive [22].

However, the advantage of vacuum microwave drying was to speed up drying process, to increase mass transfer by an increased pressure gradient between inner and outer layers of berries and to maintain drying process at low temperature [23].

Of course, for better water evaporation an important role has pretreatment method of berries before drying, too. Therefore it was significant to analyse influences of various pre-treatment methods and drying condition on volatile compound presence in cranberries.

TABLE II
THE SIGNIFICANCE (P-VALUES) OF ANOVA MODEL MAIN AND INTERACTION EFFECTS (*RESULT IS SIGNIFICANT AT P = 0.95, **RESULT IS SIGNIFICANT AT P = 0.99)

Substance	Factor: cultivar	Factor: pre-treatment of berries	Factor: drying method	Cultivar* pre-treatment of berries	Cultivar* drying method	Pre-treatment of berries* drying method	3 factor interaction
4-penten-2-ol	0.00**	0.00**	0.00**	0.00**	0.00**	0.31	0.02*
Nonanoic acid	0.69	0.53	0.14	0.38	0.53	0.93	0.31
Acetic acid	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**
Benz-aldehyde	0.03*	0.72	0.74	0.33	0.11	0.11	0.84
3-metyl-butan-1-ol	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**
α-1-terpineol	0.00**	0.00**	0.00**	0.00**	0.00**	0.01**	0.00**
Benzyl alcohol	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**
Phenylethyl alcohol	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**
Caprylic acid	0.33	0.03*	0.19	0.42	0.10	0.41	0.54
Pelargonic acid	0.00**	0.00**	0.71	0.00**	0.01*	0.00**	0.11
Benzoic acid	0.55	0.01**	0.04*	0.63	0.48	0.33	0.34
Hexanal	0.25	0.80	0.33	0.33	0.73	0.37	-
Metoxi-propan-2 -ol	0.10	0.39	0.94	0.54	0.30	0.05*	0.05*
α-phellandrene	0.44	0.02*	0.17	0.11	0.66	0.83	0.82
D-limonene	0.08	0.05*	0.39	0.03*	0.25	0.85	0.15
Hydroxy-butan-2-one	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**
Furfural	0.00**	0.00**	0.00**	0.00**	-	-	-
Hydroxy-2-propanamide	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**	0.00**
Methyl-propanoic acid	0.00**	0.40	0.15	0.00**	0.00**	0.00**	0.00**
Hexan-1-ol	0.05*	0.00**	0.12	0.00**	0.02*	0.05	0.01**

In the present research it was established, that there are differences in detected volatile compounds using several pre-treatment methods and dehydration in convective whether microwave-vacuum driers (as an example the cranberry cultivar "Steven" data are shown in Fig. 2 and Fig. 3).

A three-way ANOVA analysis using IBM SPSS 20.0 was selected to investigate factor effects (substance, cultivar and drying method) and interactions among them. The analysis of variance (ANOVA) was used as statistically based technique capable of producing meaningful models on the importance of the factors studied in the experiment and possible factor interactions within the data set that may have an influence on the cranberries substances variation to be studied.

The ANOVA model has the following form:

$$y_{ijkl} = \mu + \alpha_i + \beta_j + \gamma_k + (\alpha\beta)_{ij} + (\alpha\gamma)_{ik} + (\beta\gamma)_{jk} + (\alpha \cdot \beta\gamma)_{ijk} + \varepsilon_{ijkl} \quad (1)$$

However, for interpreting ANOVA results, it is used only the higher-order term as the three-way interaction, because also it is statistically meaningful to start with higher-order term than lower-order terms, whereas lower order terms are generally main effects. Table 2 summarizes the ANOVA results.

Three-way interactions were found to be statistically significant for 4-penten-2-ol ($p = 0.024$), acetic acid, 3-metyl-butan-1-ol, α-1-terpineol, benzyl alcohol, phenylethyl alcohol, hydroxy-butan-2-one, hydroxy-2-propanamide, methyl-propanoic acid, hexan-1-ol ($p = 0.000$), as well as for metoxipropan-2-ol ($p = 0.05$). Three-way interactions for another substances was not significant at α level = 0.05

In the present experiments many new volatile compounds were detected in dried cranberries (comparing substances form Table I and Table II). In dried cranberries 20 volatile compounds were detected totally. Presence of new volatile compounds in dried cranberries mainly could be explained with the chemical and biochemical reactions occurring in berries during drying, it is related to thermal treatment.

Pelargonic acid is found naturally in almost all animal and plant species, and it is used as a nonselective, foliar-applied broad-spectrum herbicide to control annual and perennial weeds [24]. During experiments it was established, that the main pelargonic acid volatile formation occurs in berries dried in cabinet drier, mainly because longer processing at elevated temperatures and possible fertilizers presence in cultivated berries.

In fresh cranberries acetic acid practically was not detected, however acetic acid presence is established in dried cranberries. It forecast longer berries shelf life because acetic acid preservative properties and berries fungal protection. Pronounced acetic acid formation occurs in berries dried in microwave-vacuum drier especially if berries halving or perforation was used as a pre-treatment method before drying.

Benzaldehyde presence in dried cranberries was lower comparing with fresh cranberries, especially in berries dried in microwave-vacuum drier, what mainly could be explained with microwave influence on volatile compound destruction.

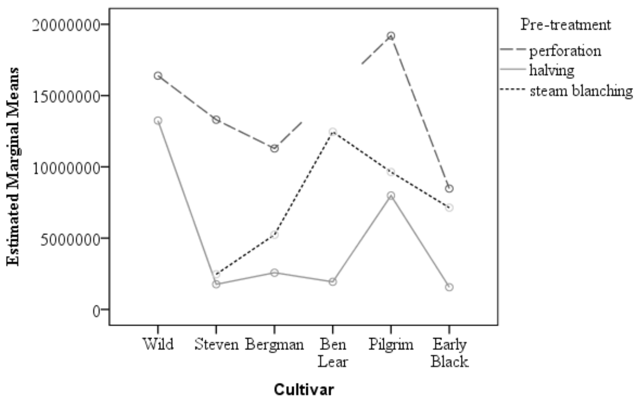

Fig. 4 Interaction of berry cultivar, pre-treatment method and drying conditions for 4-penten-2-ol presence in berries (dried in convective drier)

The presence of 4-penten-2-ol, as fruity aroma, was lower in dried cranberries than in fresh cranberries especially if berries were processed in a cabinet drier, what mainly indicate negative temperature effect on volatile compound stability. For interpretation of the acquired results the interconnection effects were studied.

The interconnection was searched for all detected volatile compounds separately. The example could be explained with 4-penten-2-ol. If berries were dried the interaction was found between three main factors, as berries pre-treatment, cultivars and drying method (Fig. 4, 5).

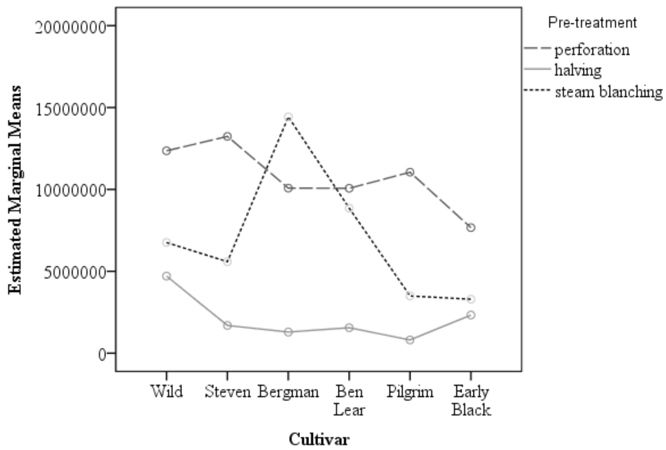

Fig. 5 Interaction of berry cultivar, pre-treatment method and drying conditions for 4-penten-2-ol presence in berries (dried in microwave-vacuum drier)

Presence of volatile compounds in perforated berries dried in convective drier (Fig. 4) was more pronounced than in perforated berries dried in microwave-vacuum drier (Fig. 5). However, the stem-blanching could be recommendable to retain the volatile compound 4-penten-2-ol in berries, especially in cultivar 'Bergman' when dried in microwave-vacuum drier.

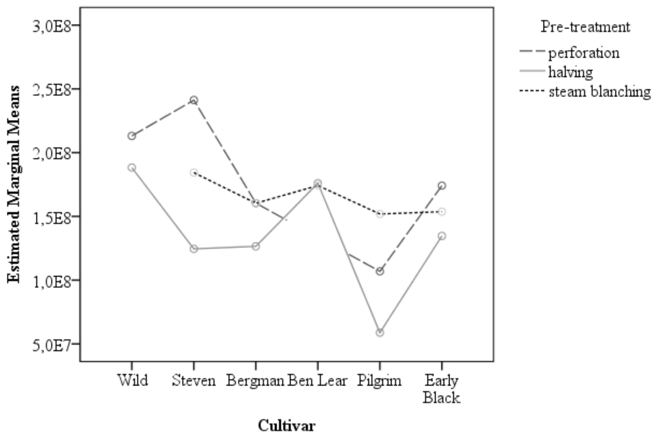

Fig. 6 Interaction of berry cultivar, pre-treatment method and drying conditions for acetic acid presence in berries (dried in convective drier)

Above it was mentioned that acetic acid formation was indicated. Higher acetic acid formation occurs in halved and perforated berries dried in vacuum-microwave drier (Fig. 6), especially if berries were steam-blanched and halved before drying.

Acetic acid formation in berries dried in convective drier was less pronounced.

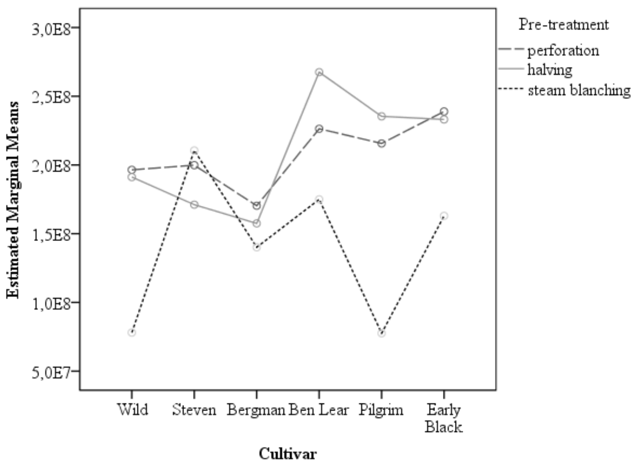

Fig. 7 Interaction of berry cultivar, pre-treatment method and drying conditions for acetic acid presence in berries (dried in microwave-vacuum drier)

Mathematical data processing enables to describe effect of interaction between three main factors as berry cultivar, berry pre-treatment method and drying conditions on volatile compound formation in berries. It is possible to analyse all three factor interaction, as influence on separate volatile compound. Therefore, in future experiments it is necessary to indicate the major volatile compounds and as a result to choose the most appropriate pre-treatment method and drying conditions.

V. Conclusion

During present research 21 various volatile compounds were detected in fresh cranberries and 20 – in dried cranberries. Many common volatile compounds were found in fresh cranberry cultivars and wild cranberries, as 4-penten-2-ol (fruity), 3-cis-hexenyl formate (melon-like and fruity notes respectively); benzaldehyde (almonds), α-1-terpineol (floral), butyric acid (rancid butter), benzyl alcohol (sweet and fruity).

Performed mathematical data processing enables to describe effect of interaction between three main factors as berry cultivar, berry pre-treatment method and drying conditions on formation of volatile compound in berries.

Acquired results demonstrate, that significant influence there is found among cultivar, pre-treatment method and drying conditions on volatile compound formation in cranberries during drying process.

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