



Effect of ripening temperature on the chemical composition of lingonberries (*Vaccinium vitis-idaea* L.) of northern and southern origin

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ABSTRACT

Lingonberries (*Vaccinium vitis-idaea* L.) from two locations, northern (69°N, 18°E) and southern (59°N, 10°E) Norway, were grown under controlled conditions in a phytotron at two temperatures (9 and 15 °C) to study the effects of the ripening temperature and origin on the chemical composition of the berries. The concentrations of phenolic compounds, sugars, and organic acids as well as the profile of volatile organic compounds (VOCs) were determined using chromatographic and mass spectrometric methods. Five anthocyanins, eleven flavonols, eight cinnamic acid derivatives, three flavan-3-ols, three sugars, three organic acids, and 77 VOCs were identified, of which 40 VOCs had not previously been reported in lingonberries. Berries from both locations, were found to have higher contents of anthocyanins and cinnamic acid derivatives when ripened at lower temperature (9 °C), compared to the higher temperature (15 °C). Lingonberries of northern origin had a different VOC profile and higher contents of anthocyanins and organic acids than berries originating from the south. Lingonberries from the northern location also had higher proportions of cyanidin-3-O-glucoside and cyanidin-3-O-arabinoside than lingonberries from the southern location. The results show that the composition of lingonberries is influenced by both the environment and the origin of the plants, with phenolic compounds mainly influenced by the growth temperature and VOCs mainly influenced by plant origin.

1. Introduction

Lingonberry (*Vaccinium vitis-idaea* L.) is an evergreen dwarf shrub native to the northern hemisphere that bears bright scarlet red berries in autumn. It has been estimated that between 130 and 390 million kilograms of edible lingonberries are ripening in Finnish forests alone, of which only around 7 % is harvested annually (Salo, 2015). Lingonberries are a traditional part of the Nordic diet, primarily consumed as jam, juices and in desserts. Lingonberries are also an important source of income for rural communities and a commodity for export (Hjalmarsson and Ortiz, 2001; Salo, 2015). Berries from the *Vaccinium* genus, have increased in popularity over the last years, attributed to their high content of a variety of bioactive compounds, dietary fibres, and

micronutrients (Kowalska, 2021), making them a valuable part of a healthy diet.

Lingonberry is generally considered as a stress-tolerant species, which can grow in areas with varying temperatures, although it prefers areas with relatively low temperatures (Hjalmarsson and Ortiz, 2001). The growth environment influences the biosynthesis of specialized metabolites in plants and affects the adaptation of plants to different growth locations by changing the accumulation of metabolites (Kissen et al., 2016; Zoratti et al., 2014). Phenolic compounds are the most widely studied secondary metabolites in lingonberries (Andersen, 1985; Bujor et al., 2018; Ek et al., 2006; Viljanen et al., 2014). Lingonberries are among the *Vaccinium* species with the highest number of volatile organic compounds (VOCs) (Sater et al., 2020). VOCs are important for

Abbreviations: Dw, Dry weight; DAD, Diode array detector; Fw, fresh weight; GC, Gas chromatography; HPLC, High-performance liquid chromatography; HS-SPME, Head space solid phase micro extraction; MS, Mass spectrometry; PA, Proanthocyanidin; PCA, Principal component analysis; RI, Refraction index; VOC, Volatile Organic compounds; DVB/CAR/PDMS, Divinylbenzene-carboxen-polydimethylsiloxane.

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the aroma and flavour of foods. However, only a few studies have assessed the contents of these compounds in lingonberries (Sater et al., 2020). It is challenging to understand the drivers of the quality traits of wild species such as lingonberries. The contents of both primary and secondary plant metabolites associated with quality are influenced by factors such as the ripening stage, genotype, and preharvest biotic and abiotic conditions (Karppinen et al., 2016; Farneti et al., 2017). The contents of phenolic compounds have been shown to vary markedly from northern to southern latitudes in other wild *Vaccinium* species (Lätti et al. 2008, Lätti et al. 2010, Åkerström et al. 2010). Earlier field studies have indicated association between temperature and the accumulation of different groups of phenolic compounds and total phenolic content in wild stands of lingonberries (Alam et al., 2016; Vyas et al., 2015; Bujor et al., 2018; Vilkickyte and Raudone, 2021b). The sugar content in lingonberries has been found to be inversely correlated with latitude (Vilkickyte and Raudone, 2021b). Even though field studies provide valuable information on the effects of environmental factors on the quality of berries, the fluctuation of biotic and abiotic conditions and

their covariation makes the estimation of the role of specific environmental factors difficult. The knowledge of temperature effects will become increasingly important due to predicted increases in mean temperatures across the globe, with the largest effects seen in Arctic and Antarctic areas (IPCC, 2021). Furthermore, no previous studies have investigated the association between the growth conditions and VOCs and organic acids in lingonberries. As phenolic compounds, VOCs, sugars, and organic acids are known to influence the taste and flavour of lingonberries, more studies are needed to highlight the key factors affecting the accumulation of these compounds.

Therefore, the aim of this study was to investigate the influence of the ripening temperature and latitudinal origin on the metabolomic profile and chemical composition of lingonberries grown under controlled conditions. Lingonberries originating from northern and southern Norway ripened at 9 and 15 °C in a phytotron to assess the effects of origin and the response to temperature on phenolic compounds, VOCs, sugars, and organic acids. This study is the first comprehensive report on the chemical quality of lingonberries in

Table 1

Concentrations of phenolic compounds (mg/100 g fw), sugars (g/100 g fw) and organic acids (g/100 g fw) in lingonberries from two locations in Norway (north and south) ripened in a phytotron at 9 and 15 °C and harvested from the wild^a. Results from ANOVA (p values) of significant differences between samples from the phytotron experiment^b.

	Significance (P-values) ^b			North		South		North		South	
	T	O	T × O	9 °C	15 °C	9 °C	15 °C	Wild stands	Wild stands	Wild stands	Wild stands
Cyanidin-3-O-galactoside	0.000	0.038	0.006	61.1 ± 1.8 bc	44.2 ± 1.4c	71.3 ± 3.3b	42.4 ± 3.8c	101.6 ± 11.1 a	65.4 ± 11.0b		
Cyanidin-3-O-glucoside	0.018	0.000	0.274	4.1 ± 0.2 abc	5.0 ± 0.4 a	2.8 ± 0.3c	3.2 ± 0.4 bc	3.1 ± 0.3c	4.5 ± 1.0 ab		
Cyanidin-3-O-arabinoside	0.004	0.000	0.179	18.4 ± 1.1 a	13.7 ± 1.3 bc	9.0 ± 1.1 d	6.8 ± 2.2 d	14.3 ± 2.4 ab	10.0 ± 0.7 cd		
Cyanidin-3-O-pentoside	0.000	0.002	0.271	1.4 ± 0.1 a	0.9 ± 0.1 bc	1.0 ± 0.1b	0.6 ± 0.2c	1.4 ± 0.1 a	0.9 ± 0.1 bc		
Cyanidin-3-O- (acetyl)glucoside	0.041	0.044	0.506	0.8 ± 0.0	0.3 ± 0.0	0.3 ± 0.5	0.1 ± 0.1	0.5 ± 0.0	0.1 ± 0.1		
Total anthocyanins	0.000	0.016	0.067	85.9 ± 2.6b	64.2 ± 2.9 bc	84.5 ± 2.0b	53.1 ± 5.8c	120.9 ± 12.8 a	80.9 ± 12.0b		
Quercetin-3-O-galactoside	0.090	0.065	0.128	5.1 ± 0.4b	5.0 ± 0.3b	6.9 ± 0.3b	5.2 ± 1.5b	17.4 ± 5.2 a	5.5 ± 0.6b		
Quercetin-3-O-glucoside	0.216	0.363	0.982	0.8 ± 0.1b	0.9 ± 0.1b	1.4 ± 0.0b	1.2 ± 0.3b	3.0 ± 1.0 a	1.2 ± 0.3b		
Quercetin-3-O-xyloside	0.839	0.392	0.886	1.0 ± 0.1	1.0 ± 0.1	1.3 ± 0.1	1.2 ± 0.2	1.3 ± 0.1	1.3 ± 0.2		
Quercetin-3-O-arabinoside	0.000	0.000	0.028	6.8 ± 0.3b	6.4 ± 0.2b	8.5 ± 0.5b	6.4 ± 1.7b	12.8 ± 2.2 a	7.8 ± 0.8b		
Quercetin-3-O-arabinofuranoside	0.398	0.003	0.165	0.4 ± 0.0b	0.4 ± 0.0b	0.7 ± 0.2 a	0.4 ± 0.0b	0.6 ± 0.0 ab	0.5 ± 0.1b		
Quercetin-3-O-rhamnoside	0.399	0.009	0.419	7.2 ± 0.1 ab	6.7 ± 0.6 ab	10.6 ± 1.2a	10.5 ± 0.5 a	7.4 ± 2.0 ab	3.8 ± 2.6b		
Quercetin-3-O-(HMG)-pentoside 1 ^c	0.039	0.161	0.148	0.6 ± 0.1 a	0.5 ± 0.1 a	0.3 ± 0.0b	0.2 ± 0.1b	0.3 ± 0.1b	0.3 ± 0.0b		
Quercetin-3-O-(HMG)-pentoside 2 ^c	0.006	0.006	0.058	0.4 ± 0.0	0.4 ± 0.0	0.5 ± 0.1	0.4 ± 0.0	0.3 ± 0.1	0.2 ± 0.1		
Kaempferol-3-O-rhamnoside	0.458	0.000	0.567	0.2 ± 0.0b	0.3 ± 0.0b	0.1 ± 0.0b	0.2 ± 0.0b	0.3 ± 0.0b	0.7 ± 0.3 a		
Quercetin-3-O-(HMG)-rhamnoside ^c	0.347	0.001	0.694	7.5 ± 0.6	6.9 ± 0.7	5.6 ± 3.3	5.5 ± 5.3	5.9 ± 3.1	4.6 ± 4.3		
Kaempferol-3-O-(HMG)-rhamnoside ^c	0.132	0.000	0.515	0.3 ± 0.0 ab	0.4 ± 0.0 a	0.2 ± 0.1 bc	0.2 ± 0.0 bc	0.0 ± 0.0c	0.0 ± 0.0c		
Total flavonols	0.298	0.193	0.617	30.4 ± 1.7b	28.7 ± 0.8b	36.1 ± 9.0b	31.3 ± 3.9b	49.2 ± 3.9 a	25.8 ± 7.3b		
Ferulic acid-hexoside 1	0.273	0.619	0.699	4.8 ± 0.1	3.9 ± 0.2	4.3 ± 1.2	3.9 ± 0.9	3.5 ± 0.2	1.5 ± 0.2		
Ferulic acid-hexoside 2	0.000	0.729	0.752	8.3 ± 0.7 a	3.4 ± 0.3 bc	8.4 ± 2.5 a	3.9 ± 0.9b	4.6 ± 0.7b	0.8 ± 0.1c		
Coumaroyl iridoid	0.323	0.949	0.253	1.2 ± 0.2	1.3 ± 0.2	1.6 ± 0.9	0.9 ± 0.4	0.8 ± 0.9	1.1 ± 0.8		
Caffeic acid hexoside 1	0.047	0.034	0.355	2.0 ± 0.1a	1.5 ± 0.1b	1.7 ± 0.2 ab	1.4 ± 0.3b	0.9 ± 0.1c	0.8 ± 0.2c		
Caffeic acid hexoside 2	0.001	0.003	0.081	2.1 ± 0.0 a	1.3 ± 0.1b	1.4 ± 0.4b	1.0 ± 0.1 bc	0.7 ± 0.1 cd	0.5 ± 0.1 d		
Coumaric acid – hexoside	0.527	0.021	0.708	3.7 ± 0.1	4.4 ± 0.2	6.2 ± 2.1	6.3 ± 1.6	5.7 ± 1.1	4.2 ± 0.3		
Caffeoylquinic acid (chlorogenic acid)	0.919	0.011	0.767	0.7 ± 0.2b	0.8 ± 0.3b	2.0 ± 1.0 ab	1.8 ± 0.5b	6.0 ± 3.1 a	1.3 ± 0.1b		
Sinapic acid hexoside	0.978	0.052	0.029	0.1 ± 0.0 ab	0.1 ± 0.0 ab	0.2 ± 0.0 a	0.1 ± 0.0 a	0.1 ± 0.0 ab	0.1 ± 0.0b		
Total cinnamic acid derivatives	0.050	0.361	0.951	23.1 ± 0.8 a	17.0 ± 0.9 ab	25.8 ± 8.0 a	19.4 ± 4.4 ab	22.4 ± 4.0 a	10.4 ± 1.1b		
Catechin	0.633	0.102	0.894	48.2 ± 0.9	44.5 ± 2.0	58.5 ± 25.6	33.9 ± 16.5	45.5 ± 15.6	26.1 ± 7.9		
Procyanidin a2	0.004	0.333	0.011	41.4 ± 4.3 ab	38.2 ± 7.5 ab	50.0 ± 12.1ab	40.7 ± 8.6 a	57.1 ± 2.3 a	34.1 ± 13.6b		
Procyanidin b2	0.919	0.584	0.654	25.2 ± 0.9	21.5 ± 0.2	24.3 ± 6.9	18.5 ± 6.6	19.2 ± 5.6	14.1 ± 3.4		
Total proanthocyanidins	0.269	0.202	0.419	497 ± 17 ab	473 ± 7 ab	651 ± 116 ab	510 ± 73 ab	401 ± 97 ab	269 ± 47b		
Sucrose	0.021	0.001	0.748	0.6 ± 0.0 a	0.5 ± 0.0 ab	0.5 ± 0.1 abc	0.4 ± 0.1 bcd	0.3 ± 0.1 d	0.3 ± 0.1 cd		
Glucose	0.055	0.567	0.314	2.8 ± 0.1	3.0 ± 0.0	2.8 ± 0.2	3.2 ± 0.4	3.0 ± 0.3	3.3 ± 0.4		
Fructose	0.011	0.000	0.026	2.4 ± 0.1c	2.4 ± 0.1c	2.6 ± 0.2 bc	3.1 ± 0.1 ab	2.6 ± 0.3c	3.2 ± 0.1 a		
Total sugars	0.073	0.143	0.169	5.9 ± 0.3	6.0 ± 0.1	5.9 ± 0.4	6.7 ± 0.5	5.9 ± 0.7	6.9 ± 0.4		
Citric acid	0.169	0.000	0.038	2.5 ± 0.1 a	2.4 ± 0.0 a	1.8 ± 0.0 ab	2.0 ± 0.1 ab	1.4 ± 0.3b	2.1 ± 0.6 ab		
Malic acid	0.676	0.001	0.003	0.1 ± 0.0 ab	0.1 ± 0.0 a	0.1 ± 0.0 ab	0.0 ± 0.0b	0.0 ± 0.0 ab	0.1 ± 0.0 a		
Quinic acid	0.016	0.062	0.391	1.9 ± 0.1 a	1.6 ± 0.1 ab	1.7 ± 0.2 ab	1.5 ± 0.1b	1.5 ± 0.1b	1.1 ± 0.2c		
Total organic acids	0.234	0.000	0.120	4.5 ± 0.2 a	4.1 ± 0.1 ab	3.5 ± 0.2 bc	3.6 ± 0.2 bc	3.0 ± 0.2c	3.2 ± 0.7c		

^a All concentrations are mean values ± standard deviation of three samples, except in the northern wild stand with four samples. Anthocyanins were quantified as mg/100 g fresh weight (fw) equivalents of cyanidin-3-O-galactoside at 520 nm, flavonol glycosides as quercetin-3-O-rutinoside at 360 nm, cinnamic acid derivatives as chlorogenic acid at 320 nm, and individual flavan-3-ols as catechin at 280 nm. Total proanthocyanidins was determined spectrophotometrically by the DMAC method. For the other compound groups, total content is the sum of individual compounds. Different letters (a-c) indicate significant differences (p < 0.05) between the samples as determined by the Tukey's HSD test.

^b ANOVA with the factors temperature (T) and origin (O) and their interaction (T × O).

^c HMG = Hydroxy-3-methylglutaryl.

relation to temperature and place of origin.

2. Results and discussion

2.1. Phenolic compounds

2.1.1. Phenolic composition

A total of 27 phenolic compounds were tentatively identified and quantified in lingonberries by comparison of UV and MS spectra to previously published reports of lingonberries (Table 1 and Table S1, Supporting information). The identification and contents of the major anthocyanins, flavonols and cinnamic acid derivatives (CADs) were comparable to previous analyses in lingonberries (Andersen, 1985; Ek, Kartimo, Mattila, & Tolonen, 2006; Hokkanen, Mattila, Jaakola, Pirttilä, & Tolonen, 2009; Kelanne et al., 2019; Lee & Finn, 2012; Vollmannova et al., 2009). However, earlier as many as 63 phenolic compounds have been identified in lingonberries, although several only in small quantities (Bujor et al., 2018). The five anthocyanins identified in the present study were all glycosides of cyanidin, and the flavonols were largely glycosides of quercetin. Eight CADs were quantified in the lingonberries. The total concentrations of anthocyanins, flavonols, and CADs were 53–121, 26–49, and 10–26 mg/100 g fresh weight (fw), respectively (Table 1). The contents of only three flavan-3-ols were accurately quantified with the applied HPLC-DAD method due to low molar absorptivity and coelution with other phenolic compounds. The total concentration of flavan-3-ols and their oligomers and polymers, that is, proanthocyanidins (PAs), determined by a spectrophotometric method, varied from 269 to 651 mg/100 g fw, confirming earlier research identifying PAs as the most abundant group of phenolic compounds in lingonberries (Dudonne et al., 2015; Hellström et al., 2009; Kylli et al., 2011).

2.1.2. Influence of temperature and origin on the phenolic profile

Lingonberries grown at 9 °C had significantly higher contents of anthocyanins and cinnamic acid derivatives than berries grown at 15 °C (Table 1). Previous field studies of wild lingonberries have indicated that both the leaves and berries of lingonberries accumulate higher amounts of anthocyanins, phenolic acids and flavonols and lower contents of flavan-3-ols and proanthocyanidins at lower growth temperatures (Vilkickyte and Raudone, 2021b; Vyas et al., 2015). However, these results were limited by the temperature in the local settings as the study by Vyas et al. (2015) was performed under lower temperatures and in a narrow temperature range (3.9–6.4 °C), while the temperature in the study by Vilkickyte and Raudone (2021b) was higher (~18 °C) during the growth season. An increase in the synthesis of phenolic compounds at low temperatures has been suggested as a part of the specialized protective action in berries, as they can maintain high photosynthetic rates and thereby fixate carbon (Jaakola and Hohtola, 2010). CADs are associated to the upstream synthesis of anthocyanins (Jaakola and Hohtola, 2010), and a simultaneous increase in the CADs and anthocyanins could be due to the upregulation of anthocyanin biosynthesis genes. In controlled temperature studies of bilberries (*Vaccinium myrtillus*) (grown at 12 and 18 °C), black currants (grown at 12, 18 and 24 °C) and raspberries (*Rubus idaeus*) (grown at 12, 18 and 24 °C), the optimal temperature for anthocyanin synthesis was found to be 18 °C (Remberg et al., 2010; Uleberg et al., 2012; Woznicki et al., 2016). A controlled study of cloudberries (*Rubus chamaemorus*) grown at 9, 12, 15 and 18 °C showed the highest anthocyanin content at lower growth temperatures (Martinussen et al., 2010; McDougall et al., 2011). In both lingonberries and black currants (*Ribes nigrum*), also the contents of most phenolic acids increased with decreasing temperature (Vilkickyte and Raudone, 2021b; Woznicki et al., 2016). Similarly, increases in the hydroxycinnamic acid contents in bilberries were observed at lower growth temperatures (Uleberg et al., 2012). As lingonberries ripen late in the growth season and cloudberries thrive in low-temperature areas, the increase in the synthesis of phenolic compounds could

therefore be an adaptation strategy to low temperatures. In addition to being beneficial to the plant, it has been shown that a higher content of PAs and other phenolic compounds in lingonberries give the berries a bitter taste that can be challenging by consumers (Laaksonen et al., 2016) but beneficial in relation to several health effects of the berries (Kowalska, 2021).

Among the anthocyanins, the concentration of cyanidin-3-O-glucoside was higher at 15 °C, which was the inverse of the other cyanidin-glycosides (Table 1). The profile of CADs was also affected by temperature, as the contents of ferulic acid and caffeic acid hexosides were significantly higher in lingonberries ripened at 9 °C than at 15 °C. Total flavonols were not affected by temperature and only minor effects were observed for the individual flavonols. Environmental stressors have been shown to influence the patterns of hydroxylation, methylation, and glycosylation in other plants (Alseekh et al., 2020). The hydroxylation of flavonoids has been considered a genetic trait in fruits with different species seemingly responding differently to stressors (Karppinen et al., 2016; Zoratti et al., 2014). In bilberries, decreases in both cyanidin-3-O-galactoside and cyanidin-3-O-glucoside were observed at lower growth temperatures with simultaneous increases in the concentrations of delphinidin glycosides (Uleberg et al., 2012).

Origin significantly influenced the total anthocyanin concentration in lingonberries, with the highest content found in berries of northern origin (Table 1). In lingonberries, it has previously been shown that the contents of anthocyanins, proanthocyanidins, phenolic acids and total antioxidant activity positively correlated with latitude, while total phenolics and the total flavonoid content showed less correlation (Vilkickyte and Raudone, 2021a,b; Vyas et al., 2015). The effect of origin on the anthocyanins was less significant than the variation caused by temperature. The glycosylation of the cyanidins was also affected by origin, with higher proportions of cyanidin-3-O-glucoside and cyanidin-3-O-arabinoside in berries of northern origin than in berries originating further south. In other *Vaccinium* species, such as bilberries (Lätti et al., 2008; Uleberg et al., 2012; Martz et al. 2010), and bog bilberries (*V. uliginosum*) (Lätti et al., 2010) it has also been found that northern clones produced more anthocyanins than southern clones. Latitude affects the synthesis of anthocyanins differently in different berry species; for instance, in bilberries, content of all anthocyanins increased and the proportion of cyanidin-3-O-galactoside was higher in the northern ecoregion (Uleberg et al., 2012), whereas in currants a higher proportion of several of the cyanidin-glycosides but a lower proportion of cyanidin-3-O-rutinoside in berries of the northern latitude was found (Yang et al., 2013). There was no effect of origin on the total flavonol content but significant effects were observed for many of the individual compounds including quercetin-xyloside, -arabinofuranoside -rhamnoside, -(HMG)-rhamnoside and -(HMG)-pentoside, with most compounds being more prevalent in berries from the southern origin (Table 1). In bog bilberries, the flavonol content was higher at higher latitudes compared to more southern growth places (Lätti et al., 2010). There was no effect on origin of total CADs. However, there were higher concentrations of *p*-coumaric acid hexoside and chlorogenic acid detected in lingonberries originating from the south, while the caffeic acid hexosides were more abundant in berries of northern origin. The profiles of certain phenolic acids in wild berries have also previously been shown to be affected by latitude (Vilkickyte and Raudone, 2021b). In bilberries from different origins grown under controlled conditions, the concentration of hydroxycinnamic acid derivatives was higher in bilberries from southern clones (Uleberg et al., 2012).

At the field stands, all measured phenolic groups were detected in higher concentrations in berries from the northern location compared to berries from the southern location (Table 1). There was a lower total anthocyanin content in lingonberries from the southern field stands with a higher proportion of cyanidin-3-O-glucoside. This trend could be due to both the effects of temperature and origin and their interaction, as observed in the phytotron experiment. There were also higher concentrations of total flavonols and chlorogenic acid detected in lingonberries

from the northern field stands. UV-radiation is known to influence the flavonol content as well as the content of anthocyanins in berries (Jaakola and Hohtola, 2010). Lingonberries collected from the field stands were influenced by larger fluctuations in environmental factors, including UV-radiation, temperature, and precipitation compared to berries ripened in the phytotron (Jaakola and Hohtola, 2010; Yang et al., 2013). In the northern field stands elevated levels of quercetin-3-O-galactoside, -glucoside and -arabinoside in addition to the total increase in the anthocyanins were found compared with berries ripened in the phytotron, likely due to increased UV-radiation under the open field conditions. In summary, the results indicate that both temperature and place of origin influenced the composition of phenolic compounds in lingonberries, and that temperature was the strongest contributor.

2.2. Sugars and organic acids

2.2.1. Composition of sugars and organic acids

The total sugar content in the berries varied between 5.9 and 6.9 g/100 g fw, and the content of organic acids varied between 3.0 and 4.5 g/100 g fw (Table 1). The contents of the three sugars, sucrose, glucose and fructose, were in accordance with previous reports of lingonberries, whereas the content of organic acids was higher than previously reported (Jensen et al., 2002; Kelanne et al., 2019; Mikulic-Petkovsek et al., 2012; Viljakainen et al., 2002). Some studies have reported the presence of malic or tartaric acid in lingonberries, whereas in our study, quinic acid was identified, similarly to what has been reported in Swedish lingonberries (Jensen et al., 2002).

2.2.2. Influence of temperature and origin on sugars and organic acids

Neither the total concentration of sugars nor organic acids in lingonberries ripened in the phytotron were significantly influenced by temperature (Table 1). However, higher fructose and lower sucrose contents were found in lingonberries ripened at higher temperature, particularly in berries from the south. A positive correlation between the growth temperature and total sugar concentration has been found in a field study of wild lingonberries (Vilkickyte et al., 2019). Similar results were observed in bilberries, where higher sugar concentrations were detected at 18 °C than at 12 °C (Uleberg et al., 2012). Organic acids were not significantly affected by temperature, except quinic acid, which was found at higher concentrations at 9 °C (Table 1). In accordance with our results, the temperature did not affect the total concentration of organic acids in bilberries, but a lower content of quinic acid was detected at higher growth temperatures (Uleberg et al., 2012).

The origin of lingonberries did not significantly affect the total sugar concentration in the present study, but the concentration of fructose was higher in berries of southern origin, and sucrose was slightly higher in berries of northern origin (Table 1). Similar results were found in bilberries grown under controlled conditions, where the origin of the plants did not influence the total content of sugars, apart from sucrose, which was higher in the northern clones (Uleberg et al., 2012). Lingonberries from the north had significantly higher concentrations of all three quantified acids, and there was a significant interaction between temperature and origin for citric acid and malic acid (Table 1). Uleberg et al. (2012) also reported a higher concentration of malic acid but a lower content of quinic acid in bilberries from northern clones, whereas no clear effect of clones on the total organic acid concentration was observed. Although sugars typically provide a sweet taste, the high concentration of organic acids in lingonberries may mask sweetness, and therefore, the combined content or ratio between sugars and organic acids is important for their flavour profile (Viljanen et al., 2014). This ratio is also known to influence the perceived sourness and astringency in berries (Laaksonen et al., 2016). Thus, with a higher concentration of organic acids and the same sugar concentration lingonberries from the northern location are likely to be perceived as sourer and less sweet than berries from the southern location.

No significant differences in total sugar or acid concentrations were

found in lingonberries from the wild stands in the present study compared to berries ripened in the phytotron (Table 1). There was a significantly higher content of quinic acid in lingonberries of northern origin than of southern origin in berries harvested from the field stand. As the higher total content of organic acids in berries from the northern location observed in the phytotron was not observed in berries harvested from wild stands, it indicates that other factors than temperature also affected the content of the sugars and organic acids in lingonberries.

2.3. Volatile organic compounds

2.3.1. Composition of VOCs

A total of 77 VOCs were tentatively identified in lingonberries (Table 2). Among these compounds there were 21 aldehydes, twelve esters of which nine were acetates, seven ketones, ten alcohols, eight volatile acids, 18 terpenes, of which two were unidentified, and one furan. Among the 77 compounds, 65 were found in the phytotron samples, and 12 solely found in the samples collected from the wild stands. In an earlier study, using olfactory GC-MS, 2-methylbutanoic acid, 2-methylpropanoate, hexanal, linalool, eucalyptol, diacetyl and methyl benzoate were identified as odorants in lingonberries (Marsol-Vall et al., 2020). Of these compounds, diacetyl and 2-methylpropanoate were not identified in the present study. Anjou and von Sydow, (1967, 1969) identified 2-methylbutanoic acid as the most abundant VOC and thus considered it a key compound in lingonberries, indicating that 2-methylbutanoic acid may be important for the lingonberry aroma. However, no studies have to date determined the contribution of the volatile compounds to the aroma profile of lingonberries. In cranberries, however, the compounds with the highest odour activities and thus contributing to aroma, were aldehydes and esters (Cosme et al., 2022; Zhu et al., 2016). Since lingonberries and cranberries have similar flavour characteristics, it is likely that the same groups of compounds contribute to the aroma profile of both of these berries. The aldehydes, shown to contribute to the aroma of cranberries, were also found in high abundances in the present study. However, aroma thresholds vary for the different compounds, and high concentration does not automatically mean high contribution to aroma (Maffei, 2010). The twelve esters detected in lingonberries are thought to be among important flavour and fragrance components as esters have been shown to contribute significantly to aroma of other fruits and berries. For example, esters have been among the proposed compounds responsible for the fruity flavours of blueberries at full maturity (Sater et al., 2020).

Among the 77 VOCs identified in lingonberries, 40 had not previously been identified (Table 2) (Anjou and von Sydow, 1967, 1969; Marsol-Vall et al., 2020; Viljanen et al., 2014). Also, among studies of several other *Vaccinium* species and among studies of grapes, there has been a large variation in the VOC profiles (Sater et al., 2020; González-Barreiro et al., 2015). These variations are likely to be influenced by genetic differences, or biotic and abiotic conditions (Karppinen et al., 2016). In studies of cranberries there has been some variation of compounds identified, but high contents of benzyl compounds and terpineol are generally found (Sater et al., 2020). These compounds were also detected in lingonberries in the current study (Table 2), and the benzyl compounds have been reported in all previous studies of lingonberries (Anjou and von Sydow, 1967, 1969; Marsol-Vall et al., 2020; Viljanen et al., 2014). It is recognised that the analytical technique used to determine VOCs can influence the composition of compounds (Sater et al., 2020). The studies analysing concentrated essential oils of lingonberries (Anjou and von Sydow, 1967, 1969) obtained higher number of volatile compounds and a larger variation in the compound profile than studies using HS-SPME-GC-MS (Marsol-Vall et al., 2020; Viljanen et al., 2014). Our study using HS-SPME-GC-MS gave a larger number of VOCs than the previous studies using this technique. The present study was, however, the first investigation of whole or crushed berries. It is recognized that the sample pre-treatment with freezing, partial thawing and mashing used in the present study will influence the VOC profile of

Table 2

Volatile compounds (% normalized peak area) in lingonberries from two locations in Norway (north and south) ripened in a phytotron at 9 and 15 °C and harvested from the wild^a. Results from ANOVA (p values) of significant differences between samples from the phytotron experiment^b.

	RI Measured ^d	RI literature	Ref ^e	Significance (P- values) ^b			North		South		North	South
				T	O	T*O	9 °C	15 °C	9 °C	15 °C	Wild stands	Wild stands
Aldehydes												
Acetaldehyde	716	690	1, 2	0.004	0.000	0.010	0.004 ± 0.001b	0.005 ± 0.001b	0.014 ± 0.001ab	0.024 ± 0.008ab	–	0.041 ± 0.022a
2-Methyl propanal	811	818					–	–	–	–	0.006	–
2-Methylbutanal ^c	921	910		0.365	0.000	0.365	0.003 ± 0.000b	0.003 ± 0.001b	–	–	0.019a	0.006 ± 0.006b
3-Methylbutanal	924	914		0.340	0.424	0.563	0.001 ± 0.000	0.002 ± 0.001	0.002 ± 0.001	0.003 ± 0.005	0.01	0.006 ± 0.007
Hexanal ^c	1086	1084	1,2,3	0.361	0.000	0.075	0.147 ± 0.041bc	0.255 ± 0.071c	1.528 ± 0.517a	1.199 ± 0.656abc	0.324abc	1.31 ± 0.301ab
(Z)-3-Hexenal	1142	1139		0.065	0.000	0.061	0.030 ± 0.015	0.032 ± 0.008	0.633 ± 0.342	0.383 ± 0.326	0.046	0.156 ± 0.099
(E)-3-Hexenal	1147	1131		0.213	0.000	0.189	0.092 ± 0.034	0.109 ± 0.01	2.242 ± 1.133	1.646 ± 1.288	0.132	1.104 ± 0.594
Heptanal	1187	1186	3	0.011	0.000	0.011	0.005 ± 0.003	0.009 ± 0.001	–	–	–	0.011 ± 0.019
(E)-2-Hexenal	1205	1216		0.335	0.000	0.257	0.005 ± 0.002	0.007 ± 0.001	0.086 ± 0.042	0.064 ± 0.061	–	0.018 ± 0.025
(Z)-2-Hexenal ^c	1224	1226		0.642	0.000	0.506	0.236 ± 0.081	0.265 ± 0.051	0.964 ± 0.725	0.8 ± 0.758	–	–
Octanal	1290	1291	1,2,3	0.402	0.000	0.263	0.004 ± 0.000	0.005 ± 0.000	0.003 ± 0.000	0.003 ± 0.002	–	0.005 ± 0.008
(Z)-2-Heptenal	1328	1318	1	0.052	0.001	0.254	0.008 ± 0.001	0.014 ± 0.003	0.016 ± 0.007	0.018 ± 0.01	0.007	–
Nonanal	1397	1397	1,3	0.670	0.000	0.111	0.013 ± 0.000c	0.015 ± 0.002c	0.008 ± 0.002c	0.007 ± 0.004c	0.298a	0.083 ± 0.013b
(E,E)-2,4- Hexadienal ^c	1405	1407	3	0.468	0.000	0.468	–	–	0.015 ± 0.007ab	0.012 ± 0.009ab	0.029a	0.009 ± 0.002ab
2,4-Hexadienal	1412	1406		0.749	0.000	0.995	0.094 ± 0.017a	0.097 ± 0.023a	0.029 ± 0.019b	0.032 ± 0.019b	–	0.008 ± 0.003b
(E)-2-Octenal	1441	1430	3	0.466	0.181	0.154	0.005 ± 0.001b	0.007 ± 0.001b	0.013 ± 0.009b	0.007 ± 0.003b	0.035a	0.015 ± 0.005b
2-Furaldehyde	1475	1467	1, 3	0.602	0.000	0.602	0.003 ± 0.001	0.003 ± 0.002	–	–	–	–
(E,E)-2,4- Heptadienal ^c	1477	1508	3	0.395	0.000	0.023	–	0.001 ± 0.001b	0.004 ± 0.002b	0.004 ± 0.002b	0.021a	0.014 ± 0.004a
Benzaldehyde ^c	1540	1529	1,2,3	0.238	0.594	0.508	0.006 ± 0.001	0.01 ± 0.002	0.005 ± 0.000	0.018 ± 0.023	0.006	0.006 ± 0.002
(E)-2-Nonenal	1549	1542					–	–	–	–	0.016a	0.009 ± 0.002b
p-Menth-1-en-9-al	1636	1629					–	–	–	–	–	0.001 ± 0.001
Acetate esters												
Methyl acetate	832	827		0.481	0.603	0.309	0.001 ± 0.000	0.004 ± 0.005	0.002 ± 0.000	0.002 ± 0.001	0.008	0.004 ± 0.001
Ethyl acetate	893	885	1,2,3	0.521	0.937	0.052	0.012 ± 0.000b	0.011 ± 0.002b	0.01 ± 0.002b	0.012 ± 0.001b	0.032a	0.027 ± 0.007a
Hexyl acetate ^c	1274	1271		0.132	0.000	0.026	0.045 ± 0.007a	0.069 ± 0.021ab	0.009 ± 0.005b	0.004 ± 0.002b	–	0.022 ± 0.039ab
(E)-3-Hexen-1-yl acetate	1300	1306		0.007	0.000	0.007	–	0.001 ± 0.000	–	–	–	–
(E)-3-Hexen-1-ol acetate	1317	1320					–	–	–	–	0.003	0.004 ± 0.002
(Z)-3-Hexen-1-yl acetate	1310	1316		0.661	0.699	0.029	0.034 ± 0.015	0.08 ± 0.043	0.066 ± 0.064	0.035 ± 0.026	–	–
(E)-2-Hexenyl acetate	1355	1340					–	–	–	–	0.009b	0.026 ± 0.005a
(E)-2-Hexen-1-yl acetate	1328	1333		0.614	0.000	0.629	0.08 ± 0.014a	0.073 ± 0.005a	0.001 ± 0.002b	0.001 ± 0.001b	–	–
Benzyl acetate ^c	1733	1726		0.205	0.000	0.205	–	–	0.004 ± 0.003	0.002 ± 0.002	–	0.005 ± 0.008
Other esters												
Methyl butanoate	993	984	2	0.893	0.004	0.983	0.003 ± 0.000b	0.003 ± 0.000b	0.003 ± 0.001b	0.003 ± 0.000b	0.016a	0.013 ± 0.003a
Vinyl butanoate	1046	1045					–	–	–	–	–	0.002 ± 0.004
Methyl benzoate	1642	1641	1,2,3	0.025	0.000	0.165	0.007 ± 0.001bc	0.006 ± 0.001ab	0.011 ± 0.001a	0.008 ± 0.002ab	–	0.002 ± 0.003c
Ketones												

(continued on next page)

Table 2 (continued)

	RI Measured ^d	RI literature	Ref ^c	Significance (P-values) ^b			North		South			
				T	O	T*O	9 °C	15 °C	9 °C	15 °C	Wild stands	Wild stands
2,3-Butanedione	979	978	1,2,3	0.000	0.000	0.044	0.017 ± 0.001	0.04 ± 0.01	0.005 ± 0.009	0.012 ± 0.021	0.055	0.018 ± 0.017
1-Penten-3-one	1029	1022		0.604	0.000	0.663	0.005 ± 0.002	0.001 ± 0.002	0.034 ± 0.018	0.033 ± 0.021	0.015	0.024 ± 0.021
Acetoin	1293	1287		0.001	0.009	0.989	0.003 ± 0.000	0.006 ± 0.001	0.001 ± 0.001	0.004 ± 0.007	–	–
1-Octen-3-one	1300	1304		0.006	0.002	0.016	0.001 ± 0.002	0.004 ± 0.001	0.004 ± 0.001	0.004 ± 0.002	0.008	0.004 ± 0.004
6-Methyl-5-hepten-2-one ^c	1341	1332	1,2,3	0.961	0.258	0.724	0.005 ± 0.002b	0.005 ± 0.000b	0.005 ± 0.001b	0.005 ± 0.002b	0.015a	–
Acetophenone ^c	1672	1679	1, 3	0.588	0.000	0.033	0.018 ± 0.000bc	0.017 ± 0.002b	0.009 ± 0.002c	0.012 ± 0.004bc	0.021ab	0.030 ± 0.002a
5-Ethyl-2(5H)-furanone	1777	1754		0.428	0.000	0.428	–	–	0.018 ± 0.011	0.015 ± 0.013	0.019	0.019 ± 0.004
Alcohols												
Ethanol	943	937		0.057	0.677	0.003	0.008 ± 0.002b	0.006 ± 0.001b	0.004 ± 0.000b	0.011 ± 0.009b	0.030a	0.011 ± 0.004b
2-Methyl-3-butene-2-ol	1047	1036	3	0.000	0.000	0.832	0.005 ± 0.001	0.008 ± 0.002	–	0.003 ± 0.004	–	0.019 ± 0.033
3-Methyl-1-butanol	1215	1236	1	0.391	0.009	0.631	0.001 ± 0.001	0.001 ± 0.001	0.005 ± 0.005	0.003 ± 0.006	–	0.104 ± 0.18
1-Pentanol ^c	1256	1259	3	0.579	0.000	0.824	0.011 ± 0.002	0.012 ± 0.001	0.016 ± 0.001	0.017 ± 0.006	–	0.023 ± 0.04
(Z)-2-Penten-1-ol	1322	1322		0.591	0.000	0.079	0.002 ± 0.001c	0.004 ± 0.001c	0.008 ± 0.005c	0.007 ± 0.005c	0.146a	0.036 ± 0.003b
1-Hexanol ^c	1357	1345	3	0.193	0.000	0.086	0.096 ± 0.022a	0.133 ± 0.027ab	0.068 ± 0.005b	0.063 ± 0.031bc	0.005c	0.006 ± 0.002c
(Z)-3-Hexen-1-ol	1390	1390	1, 3	0.628	0.955	0.127	0.080 ± 0.046	0.132 ± 0.077	0.121 ± 0.066	0.094 ± 0.062	0.09	0.052 ± 0.003
1-Octen-3-ol ^c	1458	1454	3	0.996	0.542	0.830	0.007 ± 0.000b	0.007 ± 0.001b	0.007 ± 0.000b	0.007 ± 0.001b	0.011a	0.012 ± 0.000a
Ethyl hexanol	1500	1499	1	0.271	0.051	0.409	0.004 ± 0.000	0.004 ± 0.000	0.004 ± 0.001	0.005 ± 0.001	–	–
Benzyl alcohol	1896	1879	1, 3	0.107	0.437	0.944	0.093 ± 0.01	0.16 ± 0.054	0.056 ± 0.011	0.129 ± 0.122	0.082	0.102 ± 0.005
Volatile acids												
Acetic acid	1462	1448		0.131	0.012	0.589	0.008 ± 0.002ab	0.01 ± 0.001b	0.007 ± 0.002b	0.008 ± 0.001b	0.005b	0.015 ± 0.003a
3-Methylbutanoic acid	1681	1676	1	0.284	0.081	0.284	–	–	0.002 ± 0.003ab	0.008 ± 0.014ab	0.049a	0.048 ± 0.032a
2-Methylbutanoic acid	1684	1670	1	0.000	0.000	0.000	0.243 ± 0.026b	0.750 ± 0.124a	0.025 ± 0.011c	0.019 ± 0.014c	–	0.037 ± 0.065c
Pentanoic acid	1737	1743		0.098	0.001	0.877	0.007 ± 0.001ab	0.009 ± 0.002ab	0.012 ± 0.003a	0.014 ± 0.003a	0.007ab	0.002 ± 0.003b
Hexanoic acid ^c	1858	1852		0.126	0.000	0.335	0.037 ± 0.009ab	0.048 ± 0.01bc	0.067 ± 0.015a	0.07 ± 0.003a	–	0.011 ± 0.006c
Heptanoic acid	1967	1956					–	–	–	–	0.14	0.075 ± 0.045
Octanoic acid	2067	2072					–	–	–	–	0.011	0.015 ± 0.004
Nonanoic acid	2169	2165		0.590	0.000	0.024	0.132 ± 0.012a	0.119 ± 0.014a	0.072 ± 0.012b	0.093 ± 0.023ab	0.012c	0.014 ± 0.007c
Terpenes												
α-Pinene ^c	1028	1027	1,2,3	0.232	0.004	0.232	0.025 ± 0.022	0.059 ± 0.040	–	–	–	0.142 ± 0.246
Camphene	1070	1063	3	0.065	0.007	0.065	0.001 ± 0.001	0.003 ± 0.002	–	–	–	0.008 ± 0.014
β-Pinene	1105	1112	3	0.056	0.015	0.056	0.001 ± 0.001	0.007 ± 0.003	–	–	–	0.013 ± 0.023
β-Thujene	1116	1124		0.221	0.013	0.221	0.002 ± 0.003	0.004 ± 0.003	–	–	–	–
Unknown terpinene	1119						–	–	–	–	–	0.005 ± 0.009a
α-Terpinene	1180	1187					–	–	–	–	1.000	1.000 ± 0.000
D-Limonene ^c	1196	1202	1, 3	0.780	0.000	0.780	0.009 ± 0.005a	0.011 ± 0.004ab	–	–	–	0.005 ± 0.005ab
Eucalyptol ^c	1215	1204	1,2,3	0.199	0.000	0.409	0.035 ± 0.030ab	0.057 ± 0.008a	–	0.005 ± 0.008b	0.007ab	0.028 ± 0.02ab
γ-Terpinene ^c	1247	1251	3	0.330	0.001	0.330	0.006 ± 0.006b	0.011 ± 0.006b	–	–	0.166ab	0.391 ± 0.283a

(continued on next page)

Table 2 (continued)

	RI Measured ^d	RI literature	Ref ^e	Significance (P-values) ^b			North		South				North	South
				T	O	T*O	9 °C	15 °C	9 °C	15 °C			Wild stands	Wild stands
Styrene	1262	1255	1				–	–	–	–			0.012b	0.023 ± 0.003a
o-Cymene ^c	1272	1265		0.303	0.001	0.303	0.005 ± 0.005	0.01 ± 0.006	–	–			–	–
Terpinolene	1283	1280		0.236	0.002	0.236	0.001 ± 0.002b	0.003 ± 0.002b	–	–			0.023a	0.005 ± 0.005b
trans-Linalool oxide (furanoid)	1487	1471		0.000	0.000	0.131	0.044 ± 0.017a	0.025 ± 0.006ab	0.011 ± 0.001bc	0.001 ± 0.001c			–	–
Linalool ^c	1555	1550	1,2,3	0.001	0.055	0.242	0.059 ± 0.013a	0.048 ± 0.011a	0.055 ± 0.01a	0.029 ± 0.006ab			0.005ab	0.002 ± 0.002b
Terpinen-4-ol	1616	1612	3	0.122	0.000	0.122	0.034 ± 0.029	0.077 ± 0.035	0.020 ± 0.034	–			–	–
α-Terpienol	1700	1696	3	0.103	0.002	0.871	0.008 ± 0.001	0.007 ± 0.001	0.007 ± 0.001	0.006 ± 0.001			–	–
Unknown terpene 2	1775			0.000	0.000	0.000	0.012 ± 0.004a	0.006 ± 0.001b	0.001 ± 0.003c	–			–	–
Camphol	1700	1698					–	–	–	–			0.008	0.010 ± 0.003
Furans														
2-Ethyl furan ^c	958	944		0.716	0.000	0.268	0.007 ± 0.002	0.009 ± 0.001	0.015 ± 0.005	0.021 ± 0.016			0.023	0.06 ± 0.063
Internal standards														
4-Methyl-2-pentanol	1172													
Neryl acetate	1728													

^a All concentrations are mean values ± standard deviation of three samples at each temperature and from each origin in berries grown in the phytotron, one sample from berries grown in the wild in berries from the northern origin and three samples from berries grown in the wild from the southern origin. Different letters (a-c) indicate significant differences ($p < 0.05$) between the samples as determined by the Tukey's HSD test.

^b ANOVA with the factors temperature (T) and origin (O) and their interaction (T × O).

^c Identified with authentic standard compound.

^d Kovats retention index for 60 m DB-WAX column (from literature).

^e References; 1, Viljanen et al. (2014); 2, Marsol-Vall et al. (2021); 3, Anjou and von Sydow (1967).

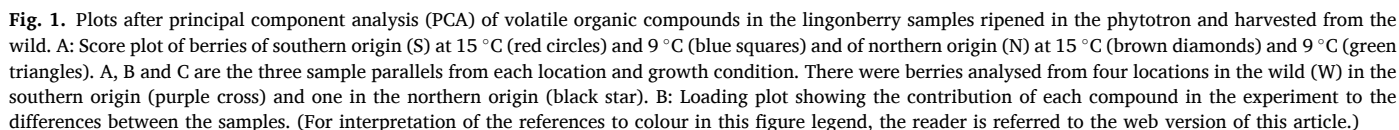
the berries. The pre-treatment was chosen to secure a consistent treatment of the berries prior to analysis and to mimic the consumer perception of aroma of lingonberries when eaten.

2.3.2. Influence of temperature and origin on the profile of VOCs

There was generally little effect of temperature on the composition of VOCs (Table 2). Temperature significantly influencing the contents of only 13 of the 65 compounds found in lingonberries ripened in the phytotron. There were significant effects of three of seven ketones and three of eleven terpenes, and the seven other belonged to the other groups. While there was no clear trend for the ketones, the three terpenes were higher at the lower growth temperature. In blackcurrants and grapes, a lower growth temperature during the last month of ripening increased the total concentration of VOCs (Marsol-Vall et al., 2018; Xie et al., 2019). The effect of temperature in these studies, however, were largely due to differences in temperature between the growth stands and could therefore also be influenced by other environmental factors than temperature (Marsol-Vall et al., 2018). In a PCA of the volatile compounds in the lingonberry samples the two first components explained 74% of the variation in the dataset (Fig. 1). There was a slight clustering and separation of the samples based on growth temperature, but the separation based on origin and samples from the wild compared to those ripened in the phytotron explained substantially more of the variation in the dataset than the temperature. Previous investigations of other species of the *Vaccinium* genus have neither managed to identify which growth factors induce changes in VOCs profile (Sater et al., 2020).

The origin of the lingonberries significantly influenced the contents of 55 of the 65 VOCs in lingonberries ripened in the phytotron, indicating a larger effect of the origin than of the temperature. Origin also affected the composition of VOCs in bilberries (Rohloff et al., 2009), highbush blueberries (*V. corymbosum*) (Du et al., 2011), blackcurrants

(Marsol-Vall et al., 2018), rabbiteye blueberries (*V. ashei*) and cranberries (*V. macrocarpon*) (Sater et al., 2020). In both blackcurrants and highbush blueberries, the same compounds were found at both northern and southern locations but with higher total concentrations in berries at the northern locations (Marsol-Vall et al., 2018; Du et al., 2011). In the PCA (Fig. 1), berries from different origin spread across PC1, which explained 51 % of the variation in the dataset, but with a large variation between the three stands in the southern origin. The variations in VOCs in berries from different places of origin may be linked to adaptations caused by genetic and environmental associations as known in several species (Karppinen et al., 2016). In blackcurrants and bilberries there was, however, a large variation between different cultivars of the same species (Marsol-Vall et al., 2018; Du et al., 2011). Among the large contributors to the variation across PC1 were the aldehydes (Fig. 1B). The aldehydes have previously been identified as key aroma compounds that could influence the flavour of *Vaccinium* berries (Sater et al., 2020). Aldehydes are mostly formed from the breakdown of lipids with C6 and C9 compounds formed through the lipoxygenase and hydroperoxide lyase pathways. In red grapes, the volatile profile is characterised by esters during early berry development, aldehydes in the middle, and alcohols being most predominant during late development (González-Barreiro et al., 2015). In addition to the higher content of the aldehydes, the berries from the southern origin had a higher content of 5-ethyl-2 (5H)-furanone. There was a large variation in profile and concentration of all terpenes between samples from the two origins with higher content in berries from the northern origin (Table 2). The terpenoids have a large variety of functions, including being important signalling molecules, often with pleasant floral and fruity scents. Previously, it has been shown that genetic differences can contribute to variation in the content of triterpenoid in lingonberries (Vilkickyte et al., 2022). There were significant interactions between temperature and origin in twelve of the VOCs, but as these were individual compounds from all groups, no



There was no major difference in the number of VOCs from lingonberries collected from the wild compared to the berries ripened in the phytotron (Table 2). There was, however, a clear difference in the compounds identified, and a separation between lingonberries harvested from wild stands compared to berries ripened in the phytotron in the PCA analysis. Berries from the wild stands clustered with a spread in the direction of PC2 compared to berries ripened in the phytotron, with PC2 explaining 23% of the variation of the dataset (Fig. 1A). Samples collected from the north and the south, however, were clustered and separated in the PC1 in a similar manner as seen for berries ripened in the phytotron. Differences between lingonberries from the northern and

the southern wild stands include a higher number of quantifiable volatiles observed in berries from the southern location.. It has previously been shown that cultivated cranberries (*V. macrocarpon*.) had a smaller number of VOCs than the wild European relative (*V. oxycoccoides*) (Sater et al. 2020). The clear differences between lingonberries collected from the wild compared to the berries ripened in the phytotron, indicate that some biotic or abiotic conditions during ripening influenced the profile of VOCs in lingonberries. Different growth conditions like light, weather, and soil composition have comparatively previously been shown to influence the volatile composition in wine grapes (González-Barreiro 2015), and are thus also likely to influence the composition of volatiles in lingonberries. The results from this study show that while there were differences in chemical composition of lingonberries from individual stands, and between berries grown in the wild or ripened in the phytotron, there also were clear differences in the VOC profile between berries from the north and south. These differences are likely to influence the perceived aroma which could give lingonberry products local flavour characteristics.

3. Conclusions

The current investigation of lingonberries showed clear effects of both the ripening temperature and the origin of the lingonberries on their metabolomic profile. Findings from the present study provide insight into how lingonberries accumulate different compounds in relation to external stressors and help us to understand the complexity of the regulation of the metabolites in these berries. Although the effects of both the temperature and place of origin were observed in many compounds, the phenolic compounds were mostly affected by temperature, and the VOCs were largely affected by origin of the berries. Altogether 40 previously undescribed VOCs were identified in lingonberries in this study. The content and composition of metabolites are important to consider, as they are crucial in plant defence against outside stressors and influence the nutritional and sensorial properties of berries. Difference in perceived aroma between lingonberries from different locations could give lingonberry products local flavour characteristics. These types of local flavoured products would be like in wine “terroir” products that are recognized to have local characteristic (González-Barreiro 2015). However, further investigation of the VOCs in relation to sensory perception are needed to assess these differences. As the biosynthesis of volatiles are complex interactional systems, further studies on both the compositional and genetic effects of different environmental conditions are needed to fully understand these interactions in lingonberries. Consequently, ongoing climate change may differentially affect the chemical profile of lingonberries in different growth locations.

4. Experimental

4.1. Chemicals

The chemicals and solvents used in this study were of HPLC-isocratic grade or higher. The water used was purified using a Milli-Q purification system (Millipore Sigma, MA, USA). Standards for HPLC and GC analyses are listed in [supplementary material](#) table s3 and included cyanidin-3-O-galactoside (Polyphenols AS, Sandnes, Norway), catechin hydrate, quercetin-3-O-rutinoside (Sigma-Aldrich, MO, USA), and chlorogenic acid (Fluka, St. Gallen, Switzerland) for analysis of phenolic compounds. Citric, malic and shikimic acids (Sigma-Aldrich, MO, USA), glucose, sucrose, and fructose (Chem Service Inc., West Chester, PA, USA), and quinic acid (Merck, Darmstadt, Germany) for analysis of sugars and organic acids. 4-Methyl-2-pentanol and neryl acetate (Sigma-Aldrich, St. Louis, MO, USA) were used as internal standards in GC-MS analysis. Hexanal, methylbutanal, (*E,E*)-2,4-hexadienal, (*E,E*)-2,4-heptadienal, benzaldehyde, 3-methyl-1-butanol acetate, hexyl acetate, ethyl octanoate, 6-methyl-5-hepten-2-one, 1-pentanol, 1-hexanol, 1-octen-3-ol, hexanoic acid, γ -terpinene, *p*-cymene, linalool, o-cymene,

2- α -pinene, *D*-limonene 2-ethyl furan, and an *n*-alkane mixture (C7–C30) were purchased from Sigma-Aldrich (St. Louis, MO, USA), 2-hexenal was purchased from Acros Organics (Antwerp, Belgium), eucalyptol, was purchased from Fluka (Steinheim, Switzerland), and acetophenone was purchased from (Chem Service Inc., West Chester, PA, USA) and used as standards in analysis of volatile organic compounds. Dimethylaminocinnamaldehyde (DMAC) (Sigma-Aldrich, MO, USA) and procyanidin A2 (Extrasynthese, Genay, France) were used for total proanthocyanidin analysis.

4.2. Plant material and experimental design

After the development of early green-stage fruits ([Fig. 2](#)), lingonberry plants were collected with intact root systems along with native soil from three wild stands (replicates) in southern (59°4N, 10°5 E, Ås) and northern (69°1N, 18°6 E, Tromsø) Norway in the 2020 growth season. The three wild stands chosen at each location were areas with many plants with fruits set within one location but with some geographical spread ([Table s2](#), [Supporting information](#)). The locations in northern and southern Norway were chosen due to a large variation in temperature during the growth season to evaluate whether the lingonberries had made a natural adaptation to temperature. The mean temperatures during the 2020 growth season in the areas chosen for testing were 9.3 °C in the north (August–September) and 17.0 °C in the south (July–August), with 3.2 mm and 4.2 mm of average daily precipitation, respectively ([Lussana, 2021](#)). The plants were transported to identical phytotron facilities in Ås (Centre for Climatic Research at the Norwegian University of Life Sciences) and Tromsø (The Arctic University of Norway and NIBIO Climate Laboratory) in their native soil. At the phytotrons, plants were placed in large growth containers and randomly assigned to one of two temperature treatments of 9 °C and 15 °C. The berries were grown under natural light with no artificial illumination and the relative humidity kept at 100 %. The day length at the period of ripening were similar at the different location, with an approximate



Fig. 2. Lingonberry plant before the collection and transport to the phytotron in Ås.

15–13 h light. The plants were watered every other day. The temperature treatments lasted for approximately 4 weeks, until most of the berries were classified as ripe, with the classification of ripeness based on surface colour being completely red. After the first harvest, berries were left to ripen for up to two weeks and picked when they were considered ripe. Within 2 h after harvest, the berries were frozen and kept frozen until analysis to maintain a coherent treatment between the samples from the two testing locations.

4.3. Methanolic extraction

Extraction of phenolic compounds, sugars and organic acids was performed following a modified version of the method described by Davik et al. (2020). Frozen lingonberries (~50 g) were milled for 15 s in a small blade mill and lyophilised for 72 h (Gamma 1–16, Christ GmbH, Osterode am Harz, Germany). The average dry matter in the lyophilised lingonberries was $16.8 \pm 0.8\%$ (Supplementary material, Table s2). A dry lingonberry sample (400 ± 10 mg) was mixed with 70 % methanol in water (v/v) (5 mL) in a vortex mixer for 15 s. This mixture was sonicated for 10 min (Ultrasonic Cleaner, VWR International, Pennsylvania, USA) and centrifuged for 10 min at $39200 \times g$ (Avanti J-26 XP Centrifuge, Beckman Coulter, California, USA). After collection of the supernatant, the insoluble plant material was re-extracted with the extraction solvent. The extractions were performed at ambient temperature (20–22 °C). Supernatants were pooled, and the volume was brought up to 20 mL with 70 % methanol in water. Extractions were performed in duplicate. The extracts were filtered through Millex HA 0.45 µm filters (Millipore Corp., Massachusetts, USA) before being transferred to HPLC vials and stored at -80 °C until analysis.

4.4. Analysis of phenolic compounds with HPLC-DAD-ESI-MS

Phenolic compounds were determined using an Agilent 1100 series HPLC system (Agilent Technologies, Waldbronn, Germany) equipped with an autosampler cooled to 4 °C, a diode array detector (DAD), and an MSD XCT ion trap mass spectrometer fitted with an electrospray ionization interface. The method has been described by Aaby et al. (2013). For analysis, 10 µL of methanolic extract was injected with separation on a Synergi 4 µm MAX RP C12 column (250 mm $\times$ 2.0 mm i.d.) equipped with a 5 µm C12 guard column (4.0 mm $\times$ 2.0 mm i.d.), both from Phenomenex (Torrance, California, USA). The mobile phase was a binary solvent system consisting of (A) formic acid/water (2/98, v/v) and (B) acetonitrile. The gradient (0–60 min with 5–60 % of B in gradual steps) eluted at a flow rate of 0.25 mL/min at 40 °C. The mass spectrometer was operated in positive and negative ion modes. Identification of phenolic compounds was performed based on their UV–vis (220–600 nm) and mass spectra and retention times relative to external standards, which were compared to previous reports of phenolic compounds in lingonberries (Bujor et al., 2018; Ek et al., 2006; Hokkanen et al., 2009; Marsol-Vall et al., 2020). Quantification was performed based on calibration curves of external standards. Anthocyanins were quantified as mg/100 g fresh weight (fw) equivalents of cyanidin-3-O-galactoside at 520 nm, flavonol glycosides as quercetin-3-O-rutinoside at 360 nm, cinnamic acid derivatives as chlorogenic acid at 320 nm, and flavan-3-ols as catechin at 280 nm.

4.5. Spectrophotometric analysis of total proanthocyanidins

The total proanthocyanidin content was quantified according to a method described by Sintara et al. (2018). The methanolic sample extracts were diluted with methanol 1/24 (v/v). The diluted sample (20 µL) was pipetted into a 96-well plate (Thermo Fisher, Massachusetts, USA), and 100 µL of 1 mg/mL dimethylaminocinnamaldehyde (DMAC) in acidic methanol (0.4 N H₂SO₄) was added. Immediately after the addition of the DMAC solution, the sample absorption was measured at 640 nm on a spectrophotometer (SpektrostarNano, BMG Labtech,

Baden-Wuerttemberg, Germany), with subsequent readings every minute for 10 min. Quantification was performed based on calibration curves of an external standard of procyanidin A2 using the average of the three last readings of the samples.

4.6. Analysis of volatile organic compounds by HS-SPME-GC-MS

VOCs were determined with a Trace 1310 gas chromatograph coupled with a TSQ 8000 EVO mass spectrometer (Thermo Scientific, Reinach, Switzerland). Extraction of volatiles was performed in a TriPlus RSH multipurpose autosampler (Thermo Scientific, Reinach, Switzerland) by using HS-SPME with a 2 cm DVB/CAR/PDMS 50/30 µm fibre (Supelco, CA, USA). Two grams of partially thawed lingonberry samples were weighed into a 20 mL HS vial. Samples were spiked with 10 µL of the internal standard mix (4-methyl-2-pentanol at 100 µg/mL and neryl acetate at 113 µg/L in methanol), and then the berries were crushed. Initially, the samples were equilibrated for 10 min at 45 °C, and the fibre was exposed to the headspace of the sample vial for 30 min at 45 °C. Volatiles were thermally desorbed in the injection port at 220 °C, and spitless injection was applied. The separation of compounds was performed with a polar capillary column (DB-WAX, 60 m $\times$ 0.25 mm $\times$ 0.25 µm: J&W Scientific, Folsom, CA, USA). Helium was used as a carrier gas with a constant flow of 1.6 mL/min. The oven was temperature-programmed from 50 °C (hold = 3 min) to 200 °C with a constant ramp of 5 °C/min (hold = 14 min). Mass selective detection was performed in the scan mode (33–300 m/z; EI (70 eV)). The interface temperature was set to 200 °C, and the ion source was set to 220 °C. Identification of the VOCs was performed by probability-based matching of the obtained mass spectra with the mass spectra from the National Institute of Standards and Technology database (NIST20) with authentic standard compounds when available. As a second criterion for the identification, Kovats retention indices (RIs) were calculated using an n-alkane mixture (C7–C30). Chromeleon (7.2.10, Thermo Scientific, Reinach, Switzerland) was used to perform peak detection, base ion detection, and peak area integration. Peak areas were normalized to the internal standards (compound area/ISTD area) previously added to each sample. Samples were analysed in quadruplicate.

4.7. Analysis of sugars and organic acids by HPLC-DAD-RI

Sugars and organic acids were determined using an Agilent 1100 series HPLC system equipped with a DAD and a refractometer index (RI) detector (Model 132; Gilson, Villiers-le-Bel, France) as described (Woznicki et al., 2017). Methanolic extracts (20 µL) were injected, and separation was performed on a Rezex ROA-Organic acid H+ (8 %) column (300 $\times$ 7.8 mm; Phenomenex, California, USA) at 45 °C with a mobile phase of 7.2 mmol/L H₂SO₄ and a flow rate of 0.5 mL/min. External standards of glucose, sucrose, fructose, and citric, malic, shikimic and quinic acids were used for quantification. The sugars were detected with an RI detector, and the organic acids were detected with DAD at 210 nm.

4.8. Statistical analyses

To assess the effect of origin and growth temperature on lingonberries grown in the phytotron, two-way analysis of variance (ANOVA) was performed. Tukey's honestly significant difference (HSD) test was performed to determine significant differences ($p < 0.05$) between all the samples. These tests were performed using the R-core package (R Core Team, 2020). To illustrate the variation among the samples in the profile of volatile organic compounds, principal component analysis (PCA) was performed with Unscrambler Software (Unscrambler®X version 10.4.1, CAMO Software AS, Oslo, Norway). All variables were weighed by 1/square root of the standard deviation before analysis.

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CRediT authorship contribution statement

M. Amundsen: Conceptualization, Methodology, Investigation, Data curation, Formal analysis, Visualization, Writing – original draft. **L. Jaakola:** Conceptualization, Methodology, Resources, Writing – review & editing. **K. Aaby:** Resources, Methodology, Writing – review & editing, Funding acquisition. **I. Martinussen:** Conceptualization, Project administration, Funding acquisition. **N. Kelanne:** Investigation, Data curation, Methodology, Writing – review & editing. **S. Tuominen:** Investigation, Data curation, Formal analysis. **O. Laaksonen:** Methodology, Writing – review & editing. **B. Yang:** Methodology, Writing – review & editing, Funding acquisition. **AL. Hykkerud:** Conceptualization, Methodology, Investigation, Writing – review & editing, Methodology, Funding acquisition.

Declaration of Competing Interest

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

Data availability

Data will be made available on request.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.foodres.2023.112738>.

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