



HS-SPME–GC–MS analysis and consumer acceptance of blackcurrant juices made with innovative and conventional dejuicing systems

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Abstract

Due to their importance for sensory perception, volatiles of blackcurrant (*Ribes nigrum* L.) juices obtained using three pressing systems in two subsequent years were assessed by HS-SPME–GC–MS. The 35 volatiles assigned comprised 15 terpenes, 10 terpenoids, six esters, two aldehydes and each one alcohol and norterpene. The aroma impact compound 4-methoxy-2-methyl-2-butanethiol (4MMB) was additionally quantitated by GC–SIM–MS after derivatization and solid phase extraction. Spiral filter press processing under exclusion of oxygen resulted in higher total concentrations of volatiles in the juices (3022 and 4865 µg/100 mL in 2019 and 2020, respectively) compared to those obtained using the conventional systems horizontal filter press and decanter (860–2122 µg/100 mL). Multivariate statistics permitted the clustering of the samples according to both, the pressing systems and year of processing and to deduce tentative marker compounds mostly contributing to their differentiation. Spiral filter pressed juices stood out for elevated levels of especially oxidation-sensitive terpenes. Levels of 4MMB ranged between 3.6 and 4.8 ng/100 mL among all samples. However, descriptive sensory analyses ($n=15$ trained panelists) found only insignificant differences among the descriptor intensities comparing blackcurrant juices and nectars produced thereof for sensory analyses, each produced with the aforementioned three technologies. By contrast, consumer acceptance and preference tests ($n=51$ consumers) showed a significant preference of the decanter made juice and the nectar derived thereof, accompanied by low ratings for the spiral filter pressed juices.

Keywords Blackcurrant volatiles · Juice processing · Spiral filter press · Oxidation · Sensory analysis

Abbreviations

Dec	Decanter
EI	Electron impact
GC–MS	Gas chromatography-mass spectrometry
HCA	Hierarchical cluster analysis
HFP	Horizontal filter press
HS-SPME	Headspace solid phase microextraction

LSD	Least significant difference
LRIs	Linear retention indices
4MMB	4-Methoxy-2-methyl-2-butanethiol
PC	Principal component
PCA	Principal component analysis
PLS-DA	Partial least squares discriminant analysis
SFP	Spiral filter press
SIM	Selected ion monitoring
SPE	Solid phase extraction

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Introduction

Blackcurrant (*Ribes nigrum* L.) belongs to the genus *Ribes* that includes about 150 species of currants and gooseberries native to central and northern Europe and northern Asia [1, 2]. The berries are rich in vitamin C, β -carotene, lutein, phenolic acids, and flavonoids, including anthocyanins that impart the typical, slightly bluish, deep black color to

the fruit [3, 4]. Besides their color, the berries are highly appreciated on worldwide markets for their pleasant flavor. The unique blackcurrant aroma is described as strong, with attributes such as ‘eucalyptus-like’, ‘cheesy-fruity’, ‘grassy’, ‘catty’, ‘pineapple-like’, ‘musty’, ‘pungent-solvent-like’, and ‘apple-like’ [5]. Previously reported profiles of volatiles consisted of more than 150 compounds, which are predominantly categorized in terpenes, oxygenated terpenoids, esters, and alcohols mainly depending on the blackcurrant variety [4, 6–9]. Oxygen-containing volatiles such as linalool, citronellol, diosphenol, *m*-cymen-8-ol, and (*Z*)-carveol described as ‘floral’, ‘minty’, ‘coniferous’ or ‘terpene-like’ prevail in the aroma profile of fresh blackcurrants [10–12]. The thiol 4-methoxy-2-methyl-2-butanethiol (4MMB) present in trace levels was found as the compound responsible for the characteristic ‘catty’ aroma [10, 11]. The compounds 4MMB (odor activity value OAV of 475, ‘sulfury, sweat’) and (*Z*)-3-hexenal (OAV 350, ‘grassy’) were among the most odor active volatiles of fresh blackcurrants, followed by ethyl butanoate (OAV 96, ‘pineapple, fruity’), 1-octen-3-one (OAV 48, ‘mushroom, metallic’), 1,8-cineole (OAV 22, ‘terpenic, forest’), hexanal (OAV 18, ‘grassy, green’), methyl butanoate (OAV 13, ‘sweat, buttery sweet’) as well as the trace constituents 2-isopropyl-, 2-isobutyl-, and 2-*sec*-butyl-3-methoxypyrazine (OAVs 13–10, ‘musty’, ‘green pepper, mushroom’, and ‘musty, earthy’, respectively) [5].

The bulk of the worldwide blackcurrant production is processed into juices, syrups, and jams [4]. Together with blackcurrant buds, which contain the same terpenes and terpenoids as the berries, they are also used to produce alcoholic beverages and essential oils for cosmetics, perfumes, and food products, but also coloring agents. A series of varieties determined for specific products with differing fruit properties like berry size or flavor intensity is available and continuously developed [4, 7].

During processing, the profile of volatiles is altered by typical unit operations like juice extraction eventually preceded by enzyme maceration, pressing, and thermal treatments, but also by post-harvest freezing when applied [3, 7, 13–15]. Apart from aroma losses, also new compounds are generated during processing, some of them being considered off-flavors [13]. For instance, blackcurrant juice concentration was shown to result in elevated levels of some monoterpenes such as terpinen-4-ol and α -terpineol, that are ultimately converted into *p*-cymene and *p*-cymen-8-ol [11].

The sensory quality of processed plant derived products is preferred by many consumers when the aroma resembles that of the raw material. Therefore, gentle processing technologies are increasingly being used in the food industry in order to maintain the sensory quality of raw material as much as possible. Losses of up to 90% of the genuine

volatiles have been reported during juice production [13, 14].

As described in our previous contribution [16], the spiral filter technology represents such a gentle technology with regard to minimizing the often deteriorative impact of oxygen exposure. The technology represents a combined modular dejuicing system consisting of a mill, an extraction chamber allowing vacuum-driven solid–liquid separation, and a collection tank, all of them being operated under exclusion of oxygen. In this previous work, the spiral filter technology was compared to two conventional systems regarding their impacts on the retention of ascorbic acid and the color determining anthocyanins during blackcurrant juice processing and storage [16]. A graphical illustration of the working principle of the two conventional systems, i.e., a decanter centrifuge or a horizontal filter press, has been made available in our previous report [17].

As particularly the aforementioned aroma-active volatiles are also considered to be crucial for blackcurrant juice quality [18–20], we now targeted at their analysis accompanied by sensory examinations, i.e., descriptive sensory profiles, consumer acceptance and preference tests. Besides juices, the widely consumed nectars, i.e., water-diluted and eventually sugar-sweetened juices were included in the study. For these purpose, frozen blackcurrants were processed at semi-industrial scale (200 kg per batch) with the innovative, oxygen excluding spiral filter press and two conventional juicing systems, i.e., horizontal filter press and decanter. The resulting juices were investigated as mentioned.

Materials and methods

Production of cloudy blackcurrant juice and nectar

The processing of blackcurrant juices using the oxygen excluding spiral filter press (VaculiQ-1000, VaculiQ, Hamminkeln, Germany) and two conventional systems, i.e., horizontal filter press (HP-L 200, Bucher, Niederweningen, Switzerland) and decanter (Z23-3, Flottweg, Vilsbiburg, Germany) was described in detail by Wagner et al. [16]. In brief, each 1200 kg frozen blackcurrants (*Ribes nigrum* L.) of mixed varieties were obtained in both years (2019 and 2020) from Obst Trautner, Gräfenberg, Germany. Each 200 kg for one of two technical repetitions per dejuicing system ($n=2$) per year were tempered to 50 °C. Subsequently, an enzyme preparation (20 mL/100 kg, Fructozym Color, Erbslöh, Geisenheim, Germany) was added and the mash was held at 50 °C for 90 min under continuous stirring. The resulting mash was subsequently processed to juice before collection in an inert atmosphere (N₂) buffer tank in case of spiral filter pressed juice and in collection tanks according

to conventional practice for the reference systems horizontal filter press and decanter. The juices were pasteurized and hot filled in one process step (78 °C) into 50 mL glass sample vessels for HS-SPME–GC–MS analyses and into 0.75 L amber glass bottles for sensory testing. Subsequently, samples for GC analyses were immediately frozen at -20 °C (both 2019 and 2020). Juice samples produced in 2020 were stored in dark environment at 4 °C until sensory analyses. By diluting the juices with tap water and by adding sucrose, nectars with a minimum fruit content of 25% as required by the European Fruit Juice Association (AIJN) [21] were processed one day before sensory testing.

HS-SPME–GC–MS analyses of blackcurrant juice volatiles

Juice volatiles were analyzed using a Trace GC equipped with a DSQII quadrupole mass spectrometer and a Trip-lus autosampler (Thermo Fisher, Dreieich, Germany) as detailed elsewhere [22]. In brief, volatiles were isolated from the headspace of the juices spiked with the internal standard 2-methyl-1-pentanol using a polydimethylsiloxane/divinylbenzene fiber (film thickness d_f =65 µm PDMS/DVB, Stable Flex[®], fiber length 1 cm, Supelco 57293-U, Sigma Aldrich, Dreieich, Germany). Chromatographic separation was achieved using a fused silica capillary column with a polar 100% polyethylene glycol stationary phase (30 m × 0.25 mm, d_f =0.25 µm ZB-Wax, Agilent J&W Columns, Santa Clara, CA, USA) and the temperature program reported previously [22]. Individual volatiles were tentatively assigned on the basis of their electron impact (EI) mass spectra and linear retention indices (LRIs) calculated according to Van der Dool and Kratz [23]. Experimental spectra and LRIs were compared to those reported in Nist 05 Libraries for XCalibur (Thermo Fisher), the NIST Chemistry WebBook [24], and literature [5, 25–35]. Semi-quantitative concentrations were expressed as µg/100 mL 2-methyl-1-pentanol equivalents.

SPE–GC–SIM–MS analyses of 4-methoxy-2-methyl-2-butanethiol (4MMB)

Analysis of the thiol 4MMB in blackcurrant juice was carried out according to the method of Kiene et al. [36], which had been based on the methods of Herbst-Johnstone et al. [37] and Coetzee et al. [38], applying the following modifications. Briefly, 100 µL of a 2 mM solution of butylated hydroxyanisole (Merck KGaA, Darmstadt, Germany) in ethanol as an antioxidant was added to 50 mL of blackcurrant juice. Subsequently, 20 µL of an ethanolic solution containing 1.8 mg/L 3SH- d_5 ([5,5,6,6,6-²H₅]-3-sulfanylhexan-1-ol, aromaLAB GmbH, Martinsried, Germany)

being used as internal standard were added. Derivatization with ethyl propiolate (ETP) and purification by SPE were performed as detailed in Kiene et al. [36]. GC–MS measurements (EI) were carried out using a 7890A GC system equipped with an MPS II robotic autosampler and a CIS 4 cooled injection system (both Gerstel, Mülheim an der Ruhr, Germany) coupled to a 5977B mass selective detector (Agilent, Santa Clara, CA, USA). Data analysis was performed with Agilent MassHunter Workstation Software (version B.08.00). Target masses for selected ion monitoring (SIM) of the 4MMB-ETP and 3SH-ETP derivatives were m/z 232, 200 and m/z 232, 187, respectively. For quantitation of 4MMB, a linear calibration was established in blackcurrant juice by plotting the concentration (ng/L) corrected by the level of 4MMB genuinely present in this sample against the ratio of the signal intensities of 4MMB-ETP/3SH-ETP.

Sensory evaluation

Sample preparation

Assessing fresh samples from both vintages with the same sensory panel was inherently impossible, as long term storage or freezing/thawing would have additionally affected the sensory perception. Therefore, sensory evaluation was exclusively conducted with fresh juice and nectar produced in 2020. The samples were brought to room temperature (20–22 °C) before pouring each 25 mL juice or nectar into ISO 3591 wine tasting glasses with a filling volume of 215 mL. The samples were labelled with random three-digit codes and presented in a randomized order to the consumer panel in a sensory testing room according to DIN EN ISO 8589 [39] as described previously [40].

Descriptive sensory analysis

For descriptive sensory analysis, four panelists specially trained for DLG (Deutsche Landwirtschafts-Gesellschaft e. V.) testing of juices and wines met in two pre-sessions, each session lasting approximately 45–60 min. In a first session they were asked to find aroma descriptors (ortho- and retronasally) for describing blackcurrant juices and nectars and to assess the references resembling the attributes. Apart from basic taste attributes ‘sweet’ and ‘sour’, the ‘astringency’ as well as six aroma descriptors with corresponding references were chosen, namely ‘fruity’ (ethyl 2-methylbutanoate; 6.3 µg/L), ‘green/grassy’ ((*Z*)-3-hexen-1-ol; 2500 µg/L), ‘floral’ (linalool; 290 µg/L), ‘sweaty’ (4-methoxy-2-methyl-2-butanethiol; 0.1 µg/L), ‘earthy’ (2-*sec*-butyl-3-methoxypyrazine; 0.4 µg/L), and ‘solvent-like’ (ethyl acetate; 1 g/L). The aforementioned aroma

Table 1 HS-SPME–GC–MS analysis of volatiles in blackcurrant juices

No	Proposed structure	CAS	Odor quality	LRI		Concentration ^a (2020)			Concentration ^a (2019)			Class ^b
				Exp	Ref	SFP	HFP	Dec	SFP	HFP	Dec	
1	ethyl acetate	141-78-6	Solvent-like, fruity ¹	886	884	15	13	15	4	2	3	Ester
2	ethanol	64-17-5	Ethanol-like ¹	930	933	18	14	16	12	8	11	Alcohol
3	methyl butanoate	80-56-8	Fruity, sweet, sweat ²	946	948	261	203	217	76	52	65	Ester
4	α -pinene	80-56-8	Forest, wood ² , pine, turpentine ³	1014	1014	37	5	0	41	11	2	Terpene, MT
5	ethyl butanoate	105-54-4	Fruity ^{1,2,4} , pineapple ⁵	1033	1030	139	114	148	129	71	105	Ester
6	camphene	79-92-5	Camphor ³	1054	1054	4	1	0	2	1	1	Terpene, MT
7	hexanal	66-25-1	Tallowy, leaf-like ¹ , green, grassy ⁴	1077	1077	3	1	0	5	2	2	Aldehyde
8	4-carene	29,050-33-7	Sweet, pungent ⁶	1119	1128	16	8	4	9	3	3	Terpene, MT
9	3-carene	13,466-78-9	Sweet, pungent ⁶	1138	1138	540	246	93	423	157	59	Terpene, MT
10	β -myrcene	123-35-3	Balsamic, must, spicy ³	1158	1158	86	20	4	60	15	5	Terpene, MT
11	α -terpinene	99-86-5	Fruity, lemon ⁷	1170	1167	140	27	9	55	17	8	Terpene, MT
12	methyl hexanoate	106-70-7	Fruity, pineapple ⁸	1185	1185	172	127	146	95	46	58	Ester
13	limonene	138-86-3	Fruity, fresh ⁹	1190	1192	257	44	25	126	35	19	Terpene, MT
14	1,8-cineole	470-82-6	Citrus, fresh ⁹	1194	1194	35	2	2	13	3	1	Terpenoid, MT
15	β -phellandrene	555-10-2	Eucalyptus ⁵ , terpenic, forest ²	1200	1200	107	75	107	179	47	54	Terpene, MT
16	β -ocimene isomer	13,877-91-3	Fragrant, fresh ⁹	1232	1233	219	98	71	160	49	41	Terpene, MT
17	γ -terpinene	99-85-4	Herb ³	1238	1238	221	45	18	88	28	11	Terpene, MT
18	<i>p</i> -cymene	99-87-6	Fruity, lemon ⁷	1265	1263	175	62	31	148	46	25	Terpene, MT
19	α -terpinolene	586-62-9	Aromatic odor ⁶	1276	1278	411	151	55	215	66	22	Terpene, MT
20	<i>cis</i> -rose oxide	4610-11-1	Lemon ³	1347	1339	11	7	8	6	7	5	Terpenoid, MT
21	methyl octanoate	111-11-5	Floral ²	1387	1387	140	40	26	50	31	12	Ester
22	nonanal	124-19-6	Waxy ⁵ , sweet, orange ⁸	1390	1390	3	3	1	3	3	3	Aldehyde
23	linalool	78-70-6	Waxy ⁵	1538	1534	26	18	11	32	16	13	Terpenoid, MT
24	β -caryophyllene	87-44-5	Floral, citrus, sweet ²	1587	1587	758	317	141	452	289	108	Terpene, SQ
25	terpinen-4-ol	562-74-3	Wood, spicy, clove-like ^{3,7}	1597	1593	411	143	96	122	112	69	Terpenoid, MT
26	γ -elemene	29,873-99-2	Spicy, clove-like ⁷	1631	1633	23	17	18	52	24	12	Terpene, SQ
27	ethyl decanoate	110-38-3	–	1636	1637	39	11	2	12	5	2	Ester
28	α -caryophyllene	6753-98-6	Fruity ¹⁰	1659	1659	363	165	71	231	155	52	Terpene, SQ
29	α -terpinyl acetate	80-26-2	Wood, spicy ¹¹	1689	1689	68	44	27	38	38	21	Terpenoid, MT
30	α -terpineol	98-55-5	Waxy, herbal, citrus, woody, floral ¹²	1693	1690	79	44	47	55	36	29	Terpenoid, MT
31	α -phellandrene-8-ol	1686-20-0	Fruity, sweet ²	1716	1718	33	22	24	23	15	14	Terpenoid, MT
32	citronellol	106-22-9	–	1767	1767	32	24	22	40	25	20	Terpenoid, MT
33	β -damascenone	23,696-85-7	Floral ² , rose ¹⁰	1817	1824	12	9	7	47	14	5	Norterpene

Table 1 (continued)

No	Proposed structure	CAS	Odor quality	LRI		Concentration ^a (2020)			Concentration ^a (2019)			Class ^b
				Exp	Ref	SFP	HFP	Dec	SFP	HFP	Dec	

34 *p*-cymen-8-ol 1197-01-9 –

35 β -caryophyllene oxide 1139-30-6 Spicy, terpenic¹³

Values represent means ($n=2$ samples from technological duplicates each year)
Volatiles were tentatively assigned based on mass spectra and linear retention indices (LRI) on a polar (ZB-Wax) stationary phase
Unless otherwise indicated, LRI reference values were taken from the NIST webbook. Further references: ¹Rychlik et al. [25], ²Jung et al. [5], ³Xiao et al. [26], ⁴Czerny et al. [27], ⁵Hempfling et al. [28], ⁶Yi et al. [29], ⁷Luo et al. [30], ⁸Du et al. [31], ⁹Pino et al. [32], ¹⁰Xia et al. [33], ¹¹Xiao et al. [26], ¹²Maikhumthod et al. [34], ¹³Kuroki et al. [35]

Dec decanter, *HFP* horizontal filter press, *IS* internal standard, *SFP* spiral filter press

^aMeans ($n=2$ samples from technological duplicate each year analyzed in analytical replicates) were expressed in μg 2-methyl-1-pentanol equivalents (IS) per 100 mL of blackcurrant juice determined using a polar (ZB-Wax) stationary phase

^bVolatiles were classified into alcohols, aldehydes, esters, terpenes (monoterpenes (terpenes, MT), sesquiterpenes (terpenes, SQ)), terpenoids (monoterpenoids (terpenoids, MT), sesquiterpenoids (terpenoids, SQ)) and nortriterpenoids

standards in concentrations 50- to 100-fold above their odor thresholds were prepared in tap water [5, 25, 27, 41], and the concentrations matching the aroma descriptors in the blackcurrant juices and nectars were used in the main session.

In the main session, 15 trained panelists (6 male, 9 female, aged between 19 and 59 years with a median at 27 years) were first trained on the descriptive attributes before tasting the three nectars, followed by the corresponding juices. During training lasting approx. 45 min, the panelists regularly participating in descriptive sensory analysis of beverages were familiarized with the blackcurrant-specific attributes. Subsequently, the panelists were asked to rank the intensity of the attributes on an unstructured continuous scale from 0 (not detectable) to 9 (strong).

The attributes ‘fruity’, ‘green-grassy’, ‘floral’, ‘sweaty’, ‘earthy’, and ‘solvent-like’ were hereby evaluated as total aroma impression (i.e., ortho- and retronasal by smelling and tasting the sample, respectively). The aforementioned references were provided to the panelists during the entire sensory evaluation. The basic tastes sweet and sour and the taste sensation astringency were also assessed in the samples.

Consumer acceptance test

The consumer acceptance of differently produced blackcurrant juices and nectars was assessed according to DIN 10974 (2008) [42] as described in detail by Vollmer et al. [40]. For panel sessions, a total of 51 university members (36 male, 15 female, aged between 19 and 55 years with a median at 24 years) were recruited and asked to assess the appearance (color), odor, taste, mouthfeel, and overall impression on a 7-point hedonic scale, ranging from ‘dislike very much’ (1 pt) over ‘neither like nor dislike’ (4 pts) to ‘like very much’ (7 pt).

Consumer preference test

Consumer preference was evaluated by the aforementioned panel ($n=51$) according to DIN 10974 (2008) [42] and DIN ISO 8587 (2006) [43] by arranging the juice and nectar samples in a forced choice ranking order in compliance with their personal preferences (most preferred, preferred, and least preferred).

Statistical analyses

Juices produced by three pressing systems (spiral filter press, horizontal filter press, and decanter) in two technical replicates ($n=2$ each pressing system) in both years 2019 and 2020 ($n=4$ in total) were examined by HS-SPME–GC–MS analyses in two analytical replicates. For concentrations

of volatiles, means and standard deviations (SD) were calculated. Significant differences of means were assessed by ANOVA ($\alpha=0.05$), following Tukey's HSD test, both conducted using JASP (Version 0.16.3.—JASP Team University of Amsterdam, Netherlands, 2022).

Principal component analysis (PCA) and a hierarchical cluster analysis (HCA) using Ward's method of agglomeration and Euclidean distances were used for unsupervised clustering of the juices. Tentative marker compounds derived from the PCA were calculated by multiplication of the absolute loadings and percentage of explained variance of the respective PC following sorting in a descending order [41].

As an alternative approach, supervised partial least squares discriminant analysis (PLS-DA) was calculated using levels of volatiles as *X*-variables and the three pressing systems as categorical *Y*-variables. Discriminative markers with absolute variable identification (VID) coefficients above 0.80 ($VID \geq |0.80|$) were derived from the PLS-DA [41]. PLS toolbox version 9.1 in MATLAB version R2021b (both MathWorks, Natick, MA, USA) was used for multivariate statistics.

Significant differences of mean descriptor intensities were tested using ANOVA ($p < 0.05$). For evaluation of consumers' acceptance and preference, hedonic values were converted to ranks. Significant differences ($p < 0.05$) were indicated by Friedman-test and a subsequent pairwise

Least Significant Difference (LSD) test based on DIN ISO 8587:2010-08 [44], calculated using Excel 2019 (Microsoft Corporation, Redmond, WA, USA).

Results and discussion

HS-SPME–GC–MS analyses of volatiles in blackcurrant juice

Total volatiles and their distribution according to their main classes

On the basis of their mass spectra and LRIs, a total of 35 blackcurrant volatiles was assigned in the juices produced by three dejuicing systems using HS-SPME–GC–MS (Table 1). In accordance with Liu et al. [45] and Jung et al. [5], 15 terpenes and 10 oxygen-containing terpenoids were the largest group of volatiles, followed by six esters, two aldehydes and each one alcohol (ethanol) and norterpene (β -damascenone).

In both years, the significantly highest concentrations of total volatiles were found in the juices produced under exclusion of oxygen using the spiral filter press (3022 and 4865 $\mu\text{g}/100\text{ mL}$ in 2019 and 2020, resp., expressed as 2-methylpentan-1-ol equivalents, Fig. 1a). Regarding the conventionally processed juices, slightly but not significantly

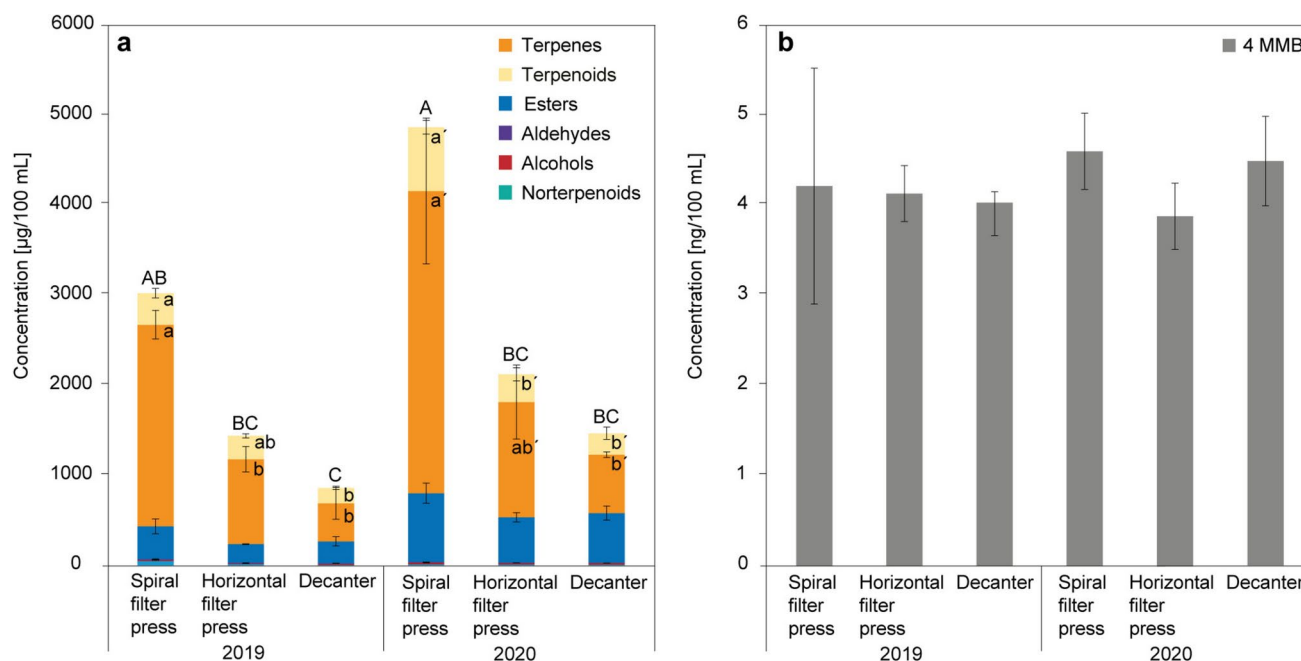


Fig. 1 Concentrations of blackcurrant volatiles in juices produced by spiral filter press, horizontal filter press, and decanter. Values represent means $\pm$ standard deviation. Significant differences of means ($p < 0.05$) within one year of processing are indicated by lower case letters, those of the total volatiles between the 2019 and 2020 samples

by capital letters. Compounds belonging to the main classes of volatiles (as 2-methyl-1-pentanol equivalents in $\mu\text{g}/100\text{ mL}$) are displayed in (a). Concentrations of 4-methoxy-2-methyl-butanethiol (4MMB) in ng/100 mL are shown in (b)

higher concentrations were found in the horizontal filter pressed samples (1437 and 2122 $\mu\text{g}/100\text{ mL}$, resp.) compared to those made by decanter (860 and 1465 $\mu\text{g}/100\text{ mL}$, resp.). Highest total volatiles were found in spiral filter pressed blackcurrant juices herein, whereas contrary observations were previously made for apple juices. Within one year of processing, total volatiles were found to be higher in horizontal filter pressed cloudy juices from red-fleshed apples than in spiral filter pressed juices [16].

Between the 2019s and 2020s samples, the highest overall concentration of total volatiles was seen in the 2020s spiral filter pressed juice (Fig. 1a). The spiral filter pressed juice in 2019 contained slightly but not significantly higher amounts of total volatiles compared to those produced with horizontal filter press in 2019 and 2020 or decanter in 2020. The decanter juices produced in 2019 showed the overall lowest contents of total volatiles among all juices.

In all blackcurrant juices, the group including terpenes and terpenoids (hereafter referred to as terpenes/terpenoids) were found in highest concentrations (594–4064 $\mu\text{g}/100\text{ mL}$), accounting for 60–84% of total volatiles isolated by HS-SPME. Among the different pressing systems, significantly highest terpene/terpenoid contents were found in spiral filter pressed juices (2589 and 4064 $\mu\text{g}/100\text{ mL}$ equaling 84 and 86% of total volatiles) potentially caused by lower oxygen exposure during processing. Considerably lower terpene/terpenoid contents were found in horizontal filter pressed juice (1202 and 1589 $\mu\text{g}/100\text{ mL}$ equaling 84 and 75%) followed by those made with decanter (594 and 885 $\mu\text{g}/100\text{ mL}$ equaling 69 and 60%, resp.).

Apart from oxygen exposure, also polarity of volatile compounds seemed to influence their enrichment or depletion in the juice matrix. Terpenes are reported to be virtually insoluble in water [46] and due to their apolar properties, large portions of terpenes are linked to cloud or pulp particles in the juice matrix [47]. As reported by Hashizume et al. [48], the concentrations of total volatiles found in apple juices increased with their levels of suspended solids. In the blackcurrant juices of our study, significantly higher cloud content in spiral filter pressed juices (23–24% cloud content) compared to the reference juices (horizontal filter press: 6%, decanter: 2%) were determined. This may additionally have promoted the transfer of terpenes/terpenoids associated with cloud particles into the spiral filter pressed juice. Volatiles categorized as monoterpenoids in Table 1 can be further classified as monoterpene alcohols (compounds 23, 25, 30, 31, 32, 34). Terpene alcohols show a higher water solubility than terpenes and thus, are enriched in the juice serum [49]. In orange juice, monoterpene alcohols were mainly found in the aqueous serum fraction [47]. Among the differently produced blackcurrant juices, all monoterpene alcohols were found in highest concentrations in spiral filter pressed juice.

The relative proportion of monoterpenes/monoterpenoids among all volatiles was highest in spiral filter pressed juice (60–61% of total volatiles), followed by the conventionally produced juices (horizontal filter press: 51%, decanter: 45–49%) and was not associated with the cloud content. Concomitantly, the relative proportion of sesquiterpenes/sesquiterpenoids (spiral filter press: 24–25%, horizontal filter press: 24–33%, and decanter: 16–20% of total volatiles) was similar in the juices from the different dejuicing systems. This observation may be explained by the lower polarity of sesquiterpenes compared to that of monoterpenes, resulting in the elevated association of monoterpenes to turbidity particles regardless of the cloud content. Similar observations were also made by Brat et al. [47], who reported an absence of sesquiterpenes in the juice serum phase, owing to their higher octanol–water partition coefficients compared to those of monoterpenes, and found 87% of sesquiterpenes liked to orange juice pulp.

Regarding terpenes/terpenoids, most interestingly, the relative contribution of oxygen-containing terpenoids increased from 14–17% over 19–22% to 27–29% in the juices obtained by spiral filter press, horizontal filter press, and decanter, respectively (values from 2019 and 2020). Decanter made juices displayed slightly elevated oxygen levels in the raw juices (see [16]), possibly resulting in the smallest concentrations of terpenes due to oxygen caused reactions yielding their oxygenated terpenoid forms. As reported in different works by Bouvier-Brown [50, 51], sesquiterpenes and oxygen-containing terpenoids are less volatile and more reactive compared to monoterpenes, resulting in their losses during thermal treatments, e.g., during juice pasteurization or even during thermal desorption after solid adsorbent pre-concentration during GC analysis.

The substance group with second highest contribution to blackcurrant volatiles were esters (Fig. 1a), accounting for 12–38% of total volatiles. Higher concentrations were found in 2020s juices (506–766 $\mu\text{g}/100\text{ mL}$) compared to 2019s juices (207–366 $\mu\text{g}/100\text{ mL}$) with slight but not significant differences between the pressing systems. Regarding their relative shares, highest proportions of esters were found in decanter made juice (28 and 38% in 2019 and 2020, resp.), followed by horizontal filter (14 and 24%, resp.) and spiral filter pressed juices (12 and 16%, resp.). In contrast to terpenes, the more polar esters have been reported to accumulate in the aqueous phase in the juice matrix rather than adsorbing to pulp or cloud particles [47, 49]. The differences in ratios between the volumes of serum and cloud particles in differently processed juices (spiral filter presses: ‘high’ cloud content of 23% and ‘low’ proportion of serum phase of 77%; decanter made juice: ‘low’ cloud content of 2% and ‘high’ proportion of serum phase of 98%) may have caused highest relative proportions of esters found in decanter

made juice due to their high share of aqueous phase. The high relative proportion of esters in decanter made juice was additionally promoted by the oxygen caused depletion of terpenes in those juices, increasing the relative proportions of the esters.

Less abundant volatiles detected by HS-SPME–GC–MS were the alcohol ethanol and aldehydes (hexanal and nonanal). Among them, merely small differences were seen between the differently produced juices (total alcohols and total aldehydes: 8 to 18 and 2 to 8 µg/100 mL, resp.).

Unsupervised classification by PCA and HCA

The concentrations of individual volatiles were used for calculating a principal component analysis (PCA). The scores and loadings of the first two principal components (PCs), describing 80% of the variance within the data are presented in Fig. 2a. The samples, representing the dejuicing systems used in 2019 (circular scores) and 2020 (squares) are separated on PC1 (62%) by the processing method and by year on PC2 (18%). HCA clustered all spiral filter pressed juices (2019 and 2020) in one main cluster, whereas a second comprised those obtained by the reference systems horizontal filter press and decanter as seen in Fig. 2a. Within the cluster comprising the reference technologies, two subclusters

were formed in dependence on the processing years (see Supplementary Information).

The tentative marker compounds for PCA-driven differentiation of the blackcurrant juices are presented in Fig. 2a', the corresponding loadings are available in the Supplementary Information. Among the marker compounds, three esters (methyl octanoate (21), ethyl decanoate (27), and methyl hexanoate (12)) were found. The four most discriminative terpenes and terpenoids exclusively comprised monoterpenes (α -pinene (4)) and monoterpenoids (citronellol (32), α -phellandren-8-ol (31), and terpinen-4-ol (25)). These C₁₀-compounds have been reported to be highly volatile [51].

All marker compounds show positive loadings on PC1, indicating their elevated concentrations in spiral filter made blackcurrant juices (see also Table 1). Among the spiral filter pressed juices, a subgroup with negative loadings on PC2 for 2020s juices consisting of methyl hexanoate (12), methyl octanoate (21), terpinen-4-ol (25), ethyl decanoate (27), and α -phellandren-8-ol (31) was found, indicating higher concentrations in those juices compared to 2019s spiral filter pressed samples. Methyl hexanoate (12) has been reported among the prevailing volatiles found in cv. Ben Lomond blackcurrant nectars [3], with high variations, fluctuating between 5 and 634 µg/kg in different varieties [5]. Also terpinen-4-ol (25) represents a characteristic

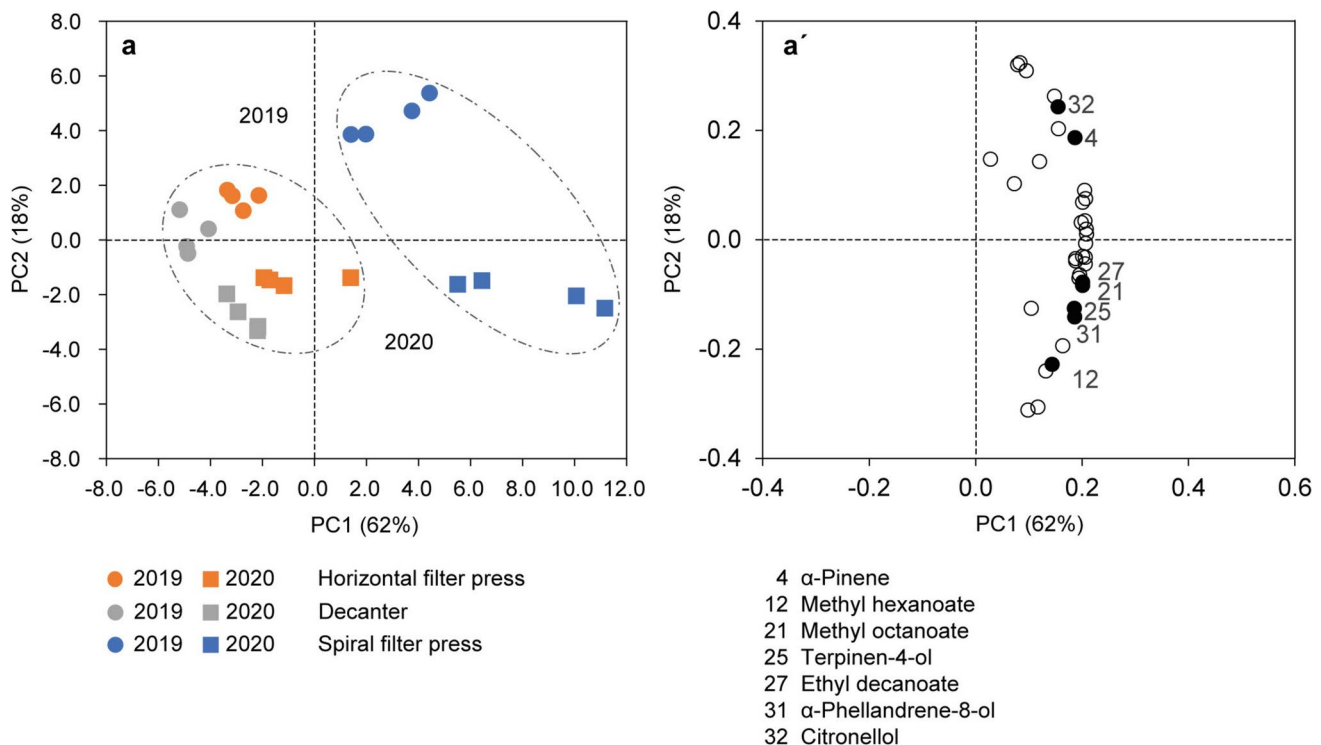


Fig. 2 PCA scores (**a**) and loadings plot (**a'**) calculated based on volatiles determined by HS-SPME–GC–MS in spiral filter press, horizontal filter press, and decanter made blackcurrant juices. The ellipses in the scores plot illustrate clusters from hierarchical cluster analysis (HCA).

Tentative marker compounds calculated by PCA are indicated by filled circles (calculations and HCA dendrogram, see Supplementary Information). Their assignment and concentrations are compiled in Table 1

blackcurrant volatile [3, 7, 9], with a high aroma impact factor and an odor described as ‘spicy’ and ‘clove-like’, found in large variations according to the blackcurrant variety (26–1588 µg/kg) [5, 10, 30]. During concentrate production, increasing levels of terpinen-4-ol were reported by Boccorh et al. [11] and Andersson and Sydow [52]. The markers also deduced from PLS-DA on the basis of their VIDs are described in more detail below.

Supervised discrimination by PLS-DA

The effects of different pressing systems on the volatiles of blackcurrant juices were further assessed using a discriminative variable selection method, i.e., partial least squares discriminant analysis (PLS-DA). The scores and loadings plots for the first two latent variables (LVs) are shown in Fig. 3.

In total, 63% of the *Y*-variance was explained by LV1 and LV2. Spiral filter pressed juices can be grouped in one separate cluster independently of the year. For juices processed using conventional horizontal filter press and decanter, clustering was less evident.

By calculating the variable identification (VID) coefficients, most discriminative volatiles in the differently produced juices were revealed. Volatiles with $VID \geq |0.80|$ are compiled in Table 2. These 22 tentative markers comprised terpenes/terpenoids, including 20 volatiles which can be divided into 12 monoterpenes and 5 monoterpenoids as

well as 2 sesquiterpenes and 1 sesquiterpenoid. Further discriminative volatiles were the esters methyl octanoate (21) and ethyl decanoate (27). The 20 markers correlated with spiral filter pressed juices included 18 terpenes/terpenoids and two esters with positive VIDs, indicating their elevated concentrations in spiral filter pressed juices. Contrarily, all 15 markers (13 terpenes/terpenoids, 2 esters) differentiating decanter made juices showed negative VIDs, as applied to the single terpene marker (β -phellandrene) discriminating horizontal filter pressed juice.

α -Pinene (4) (‘forest’, ‘woody’ [5]), has been reported as an impact compound with high contribution to blackcurrant aroma by Iversen et al. [7]. This compound already identified by PCA as discriminative marker was also deduced by PLS-DA and found in elevated levels in spiral filter pressed juices (37 to 41 vs. 0–11 µg/100 mL in the remaining samples). Due to four sites being prone to oxidation, α -pinene may be susceptible to elevated oxygen amounts and thus, spiral filter processing may have resulted in elevated levels. However, known α -pinene oxidation products such as verbenol, myrtenol, α -pinene oxide and others [53, 54] were not found (see Table 1). Camphene (6) and 3-carene (9), both chemically related to α -pinene [55] have been reported as abundant blackcurrant volatiles [56, 57] and displayed high VIDs. 3-Carene has been found in varying concentrations in different blackcurrant varieties (44–1945 µg/kg) [5].

Depending on the concentration, various aroma impressions such as ‘citrus’, ‘pine’, ‘herbal’, ‘resinous’, ‘tropical’

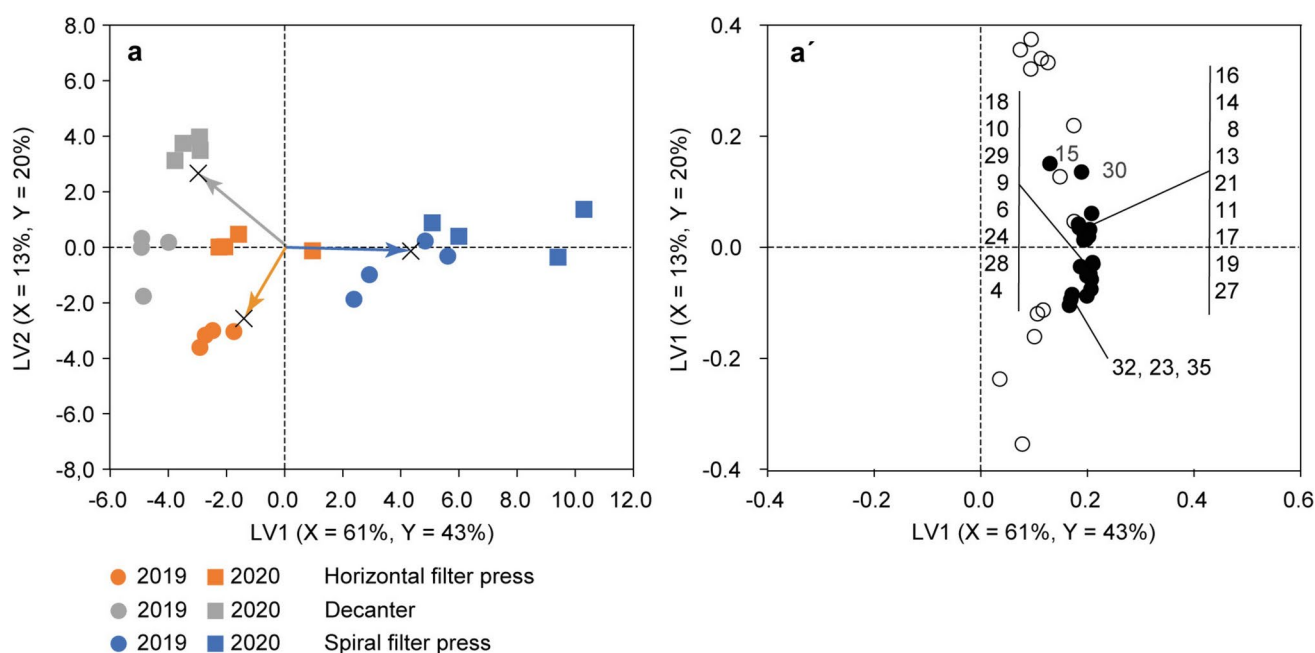


Fig. 3 PLS-DA scores (left) and loadings plot (right) calculated on the basis of the volatiles found by HS-SPME–GC–MS in differently produced blackcurrant juices in 2020 and 2019. Correlation factors for the categorical *Y*-variables are indicated by arrows, i.e., the processing

technology. Tentative marker compounds ($VID \geq |0.80|$) illustrated by black-filled circles are compiled in Table 2, their concentration and assignment in Table 1

Table 2 Discriminative volatiles ($VID \geq |0.80|$) separating blackcurrant juices produced by the dejuicing systems spiral filter press, horizontal filter press, and decanter

Spiral filter press		Horizontal filter press		Decanter	
VID	Identity (peak no.)	VID	Identity (peak no.)	VID	Identity (peak no.)
0.98	<i>p</i> -cymene (18)	− 0.81	β -phellandrene (15)	− 0.81	methyl octanoate (21)
0.97	β -myrcene (10)			− 0.83	ethyl decanoate (27)
0.97	α -pinene (4)			− 0.83	limonene (13)
0.94	β -ocimene isomer (16)			− 0.84	γ -terpinene (17)
0.94	camphene (6)			− 0.84	β -ocimene isomer (16)
0.93	3-carene (9)			− 0.84	α -terpinolene (19)
0.92	limonene (13)			− 0.84	α -terpinene (11)
0.92	β -caryophyllene (24)			− 0.87	camphene (6)
0.91	α -caryophyllene (28)			− 0.88	α -terpinyl acetate (29)
0.89	α -terpinene (11)			− 0.89	α -pinene (4)
0.88	γ -terpinene (17)			− 0.90	<i>p</i> -cymene (18)
0.87	citronellol (32)			− 0.91	β -myrcene (10)
0.87	α -terpinolene (19)			− 0.92	3-carene (9)
0.86	β -caryophyllene oxide (35)			− 0.94	caryophyllene (24)
0.84	α -terpineol (30)			− 0.96	α -caryophyllene (28)
0.83	ethyl decanoate (27)				
0.83	methyl octanoate (21)				
0.82	linalool (23)				
0.81	1,8-cineole (14)				
0.81	4-carene (8)				

VID, variable identification coefficient

‘peppery’, and ‘juniper’ have been reported [3, 5, 12, 56–58]. In contact with air, 3-carene can be oxidized yielding initially hydroperoxides, which due to certain instrumental limitations may not have been detected using HS-SPME–GC–MS. This may explain the elevated levels of this bicyclic monoterpene in juices produced under exclusion of oxygen (423 and 540 $\mu\text{g}/100\text{ mL}$ in 2019 and 2020, resp.) and smaller levels in horizontal filter (157 and 246 $\mu\text{g}/100\text{ mL}$, resp.) and decanter made juices (59 and 96 $\mu\text{g}/100\text{ mL}$, resp.). 3-Carene may also be converted to limonene (13) [59], an aroma compound also commonly found in blackcurrants [7, 60]. In spiral filter pressed juices, limonene was also found in elevated levels (126 and 257 $\mu\text{g}/100\text{ mL}$ in 2019 and 2020, resp.) as also indicated by a positive VID of 0.92 and in smaller concentrations in decanter made juices (19 and 25 $\mu\text{g}/100\text{ mL}$, resp., VID −0.83). *p*-Cymene (18), a product structurally related to limonene and which can be susceptible to oxidation [61, 62] was also found in elevated levels in spiral filter pressed juices (148 and 175 $\mu\text{g}/100\text{ mL}$, VID 0.98), especially compared to those in decanter made samples (25 and 31 $\mu\text{g}/100\text{ mL}$, VID −0.90). A possible explanation for high concentrations of limonene and *p*-cymene in spiral filter pressed juice and small levels in decanter made juice again may be the oxidation during decanter processing resulting in, e.g., limonene hydroperoxides, carveol, carvone, or limonene oxide [15, 63], and other possible compounds.

Also β -ocimene (16) (‘herbal’ [64, 65]) and linalool (23) (‘floral’, ‘citrus’, sweet’ [5]) have been reported among the most important aroma compounds in blackcurrant juice in

a study by Mikkelsen and Poll [3]. β -Ocimene is converted to linalool or linalool oxides under aerobic conditions [66], which might explain elevated levels of β -ocimene in spiral filter pressed juice. Linalool in turn may be further oxidized to linalool oxides as reported during heating of, e.g., apricot juices [15]. The formation of linalool oxides was also observed in wine at ambient conditions [67]. α -Terpineol (30) is a conversion product of limonene and linalool, the hydration is favored by low pH values as those in blackcurrant juices [67, 68]. Consecutive cyclisation reactions of α -terpineol via 1,8-terpine results in 1,8-cineole (14) [69, 70], the latter being another volatile with high VID that has been reported among the most important aroma compounds with high odor activity values in blackcurrant berries and juice [3, 5, 7].

Other oxidizable compounds found as markers with positive VIDs in spiral filter and negative VIDs in decanter made juice were β -caryophyllene (24) and α -caryophyllene (28). Again, their elevated levels in spiral filter pressed juices may result from diminished oxygen exposure during vacuum dejuicing, particularly when compared to decanter processing. However, β -caryophyllene oxide (35), an oxidation product of β -caryophyllene, displayed a positive VID coefficient and was also correlated to spiral filter pressed juice, indicating that this constituent may not only be generated during dejuicing.

The oxygen-containing terpenoid citronellol (32), also present in elevated concentrations in the spiral filter made juice, has been reported as impact compound with ‘fresh blackcurrant character’ in blackcurrant juice [3, 11].

Oxidation of citronellol has been reported to result in 1,6- and 1,7-citronellol hydroperoxides, two precursors of rose oxide (20) [71], also found in all juices produced in this work.

Targeted analysis of 4-methoxy-2-methyl-2-butanethiol (4MMB)

The trace constituent 4MMB was not detected using the HS-SPME–GC–MS method applied for the untargeted profiling analysis. Therefore, this powerful aroma compound was quantitated separately, amounting to similar concentrations of 3.9–4.6 ng/100 mL in all juices assessed (Fig. 1b). Considering the low odor threshold determined in both water and a blackcurrant-matrix of merely 0.001 µg/kg (equaling about 0.1 ng/100 mL) reported in literature [72], this would correspond to an OAV ranging from 36 to 48, thus being approx. 7.3- to 9.2-fold smaller than that reported for fresh blackcurrants (OAV 350) [5]. However, depending on the cultivar and harvesting season, a large range of OAVs of 160 to 5370 has been reported by Jung et al. [72]. The usage of frozen berries and processing may have resulted in the smaller 4MMB levels found among all juices compared to those reported for fresh berries. However, this important aroma compound still was expected to contribute to the typical blackcurrant odor of both, the juices and nectars, particular the ‘sweaty’ note perceived by the panelists (see also Fig. 4).

Descriptive sensory analysis

For both, the juices processed by different dejuicing systems and the nectars derived thereof, no significant differences were seen in the aroma descriptor intensities when comparing their sensory profiles (Fig. 4). These findings appear unexpected, because the total volatiles were significantly higher in the spiral filter pressed blackcurrant juices compared to those in the juices obtained with conventional systems.

Blackcurrant juices

For juices, slightly but not significantly higher scores for ‘green-grassy’ and ‘earthy’ were determined in the spiral filter pressed (4.6 and 4.1, resp.) compared to the conventionally produced samples (‘green-grassy’: both 3.2; ‘earthy’: 2.7–3.1) (Fig. 4a). Aroma compounds in fresh blackcurrants with ‘green-grassy’ notes reported in literature were *inter alia* (Z)- and (E)-3-hexenal, (Z)-3-hexenol, and hexanal, in addition to the trace constituents 2-isobutyl-3-methoxypyrazine and (Z)-octa-1,5-dien-3-one [5]. A potent ‘earthy’ aroma compound reported in literature is the

trace constituent 2-*s*-butyl-3-methoxypyrazine [5]. Among the volatiles detected in our HS-SPME–GC–MS profiling, α-pinene (‘forest’, ‘wood’, ‘pine’), 1,8-cineole (‘forest’), and caryophyllene (‘wood’) found as PLS-DA markers of spiral filter pressed juice may also contribute to ‘earthy’ odors in those juices. Although a large portion of spiral filter press PLS-DA markers exhibit odor qualities described as ‘fruity’ or ‘floral’, these attributes were rated at similar intensities in all juices. Slightly elevated scores for ‘sweaty-cheesy’ were seen in the decanter made juice (decanter: 4.0; horizontal filter press: 3.0; spiral filter press: 2.5), even though not reaching statistical significance at $p < 0.05$. The characteristic ‘sweaty’ note may be attributed to the thiol 4MMB (see “Targeted analysis of 4-methoxy-2-methyl-2-butanethiol (4MMB)”).

Among the basic taste attributes displayed in Fig. 4a’, ‘sour’ (7.8–8.0) was rated at high intensities, whereas ‘sweet’ was low rated (0.8–1.1). This was expected due to the high total acidity (38.6–41.7 g/L) in the blackcurrant juices [16]. The comparatively high rating of ‘astringency’ may be attributed to phenolic compounds (total phenolic compounds in the 2020s juices: 6929–7300 mg/L, see [16]).

Blackcurrant nectars

For the nectars, the attribute ‘fruity’ was still rated 4.9 to 5.1, even though the juices were diluted by the admixture of water and sugar. Low to medium intensity scores (1.1–5.9) were revealed for the remaining attributes with slight but not significantly highest scores for the decanter made nectars for the attribute ‘floral’ (4.3) followed by horizontal (3.5) and spiral filter pressed nectars (3.3) (Fig. 4b). The attribute ‘earthy’ was slightly lower rated in the decanter nectars (2.5) compared to horizontal and spiral filter pressed ones (3.2 and 3.1, resp.). Due to the dilution with water and the admixture of sugar, the mean values for ‘sweet’, ‘sour’ and ‘astringency’ were rated between 5.7 to 5.9, 3.6 to 3.8, and 1.8 to 2.2 respectively (Fig. 4b’).

By comparing the scores for juices and nectars, similar results were obtained for the perceived aroma impressions ‘fruity’ (4.6–5.3), ‘green-grassy’ (3.1–4.6), ‘floral’ (3.0–4.3), ‘sweaty-cheesy’ (2.0–4.0), ‘earthy’ (2.5–4.1), and ‘solvent-like’ (1.1–2.0) (Fig. 4a, b). By contrast, in addition to the obvious and expected differing intensities for the taste attributes ‘sweet’ and ‘sour’, the ‘astringency’ of the sweetened nectars was lower rated (1.8–2.2) compared to the undiluted juices (4.4–5.2) (Fig. 4a’, b’).

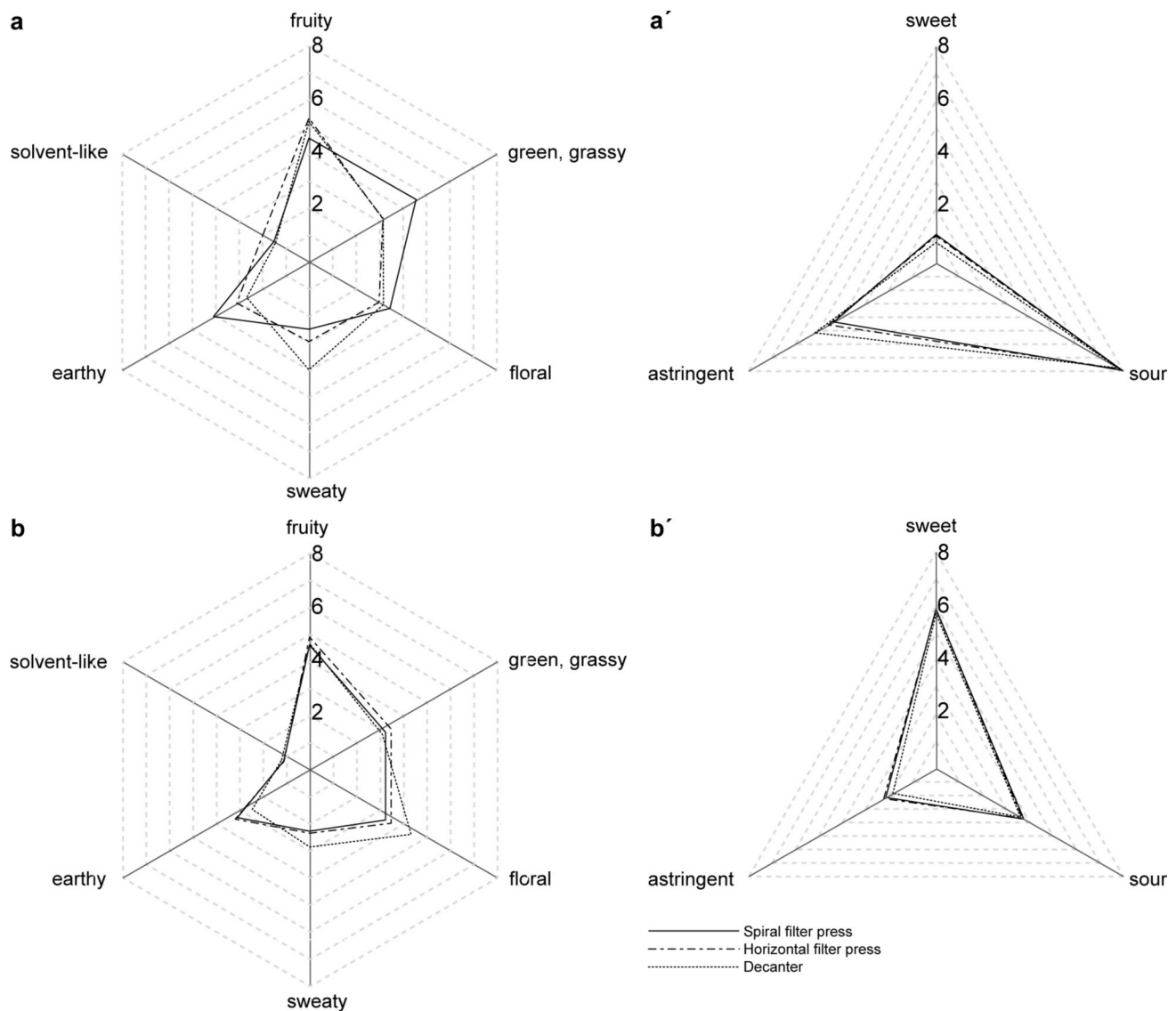


Fig. 4 Intensity scores of aroma attributes (left), the basic tastes ‘sweet’ and ‘sour’, and the taste sensation ‘astringent’ (right) of blackcurrant juices (**a** and **a'**) and nectars (**b** and **b'**) obtained by spiral filter press,

horizontal filter press, and decanter in 2020 revealed by quantitative descriptive sensory analysis ($n=15$ trained panelists). The intensity scores range from ‘not detectable’ (0) to ‘very strong’ (9)

Consumer acceptance and preference

Consumer acceptance

The frequency distributions of the hedonic scores for the attributes appearance (color), odor, taste, mouthfeel, and overall impression obtained by the consumer acceptance test for differently processed juices and derived nectars are presented in Fig. 5.

Blackcurrant juices The 51 panelists rated the visual appearance (color) for the conventionally produced juices higher [84–94% votes for ‘slightly like’ to ‘like very much’ (5–7)] compared to spiral filter pressed juices (64% votes for

5–7) (Fig. 5a). As no visible differences in juice color were detected, the differences found in the acceptance test for the appearance (color) may be particularly linked to differing turbidity and cloud content. As we had reported previously [16], turbidity and cloud content were higher in the spiral filter pressed juices (turbidity: 8778 FNU; cloud content: 23.7%) compared to horizontal filter press and decanter made juice (turbidity: 2533 and 863 FNU; cloud content: 6.2 and 1.9%, respectively, [16]).

Resultantly, significantly smaller rank sums for the attribute appearance (color) were calculated for spiral filter pressed juices (78), followed by juices obtained by

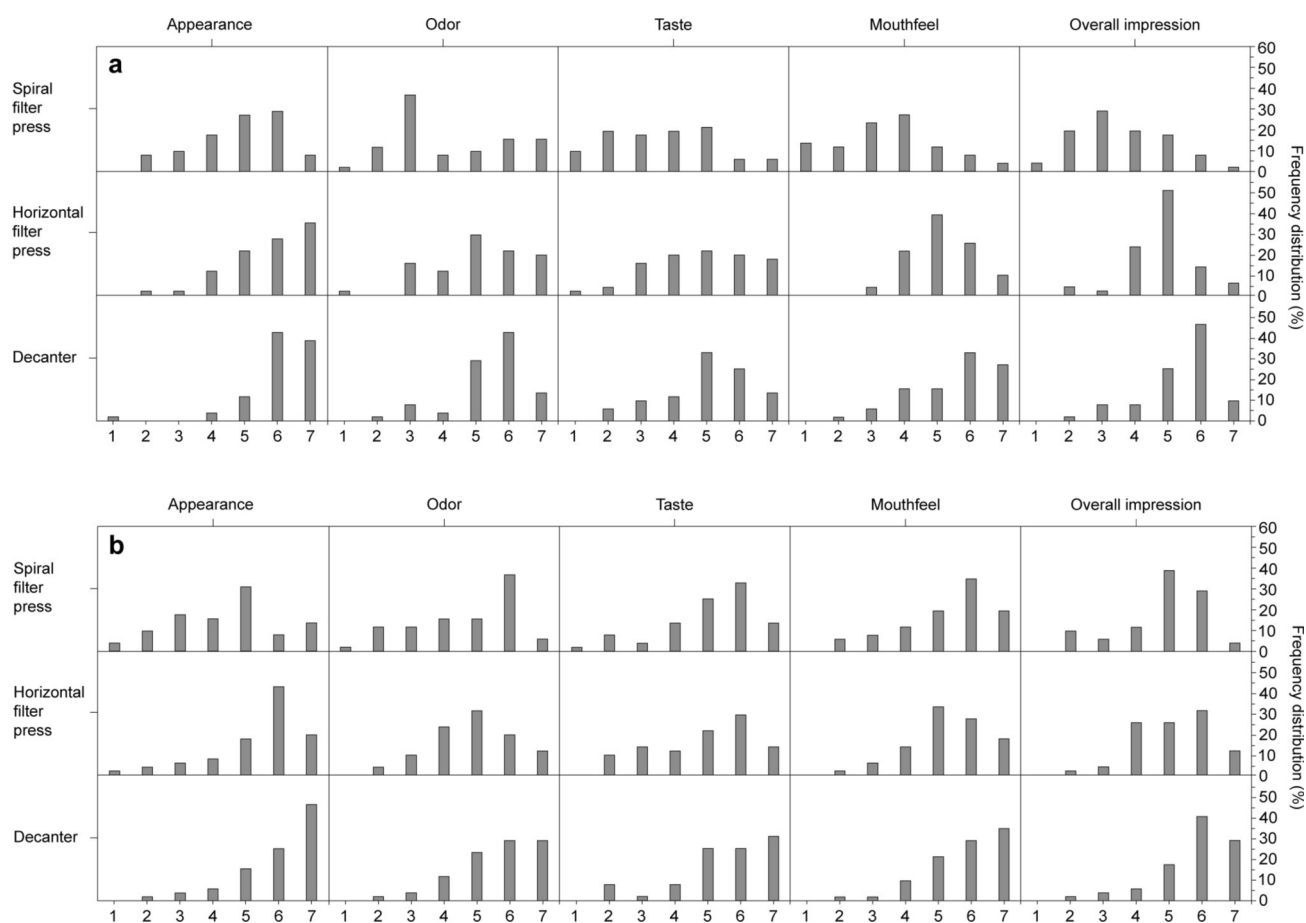


Fig. 5 Frequency distribution of hedonic scores of appearance (color), odor, taste, mouthfeel, and overall impression of blackcurrant juices (a) and nectars (b) produced by spiral filter press, horizontal filter

Table 3 Consumer acceptance and preference test of spiral filter press, horizontal filter press, and decanter made blackcurrant juices evaluated by an untrained consumer panel ($n=51$)

	Spiral filter press	Horizontal filter press	Decanter	<i>F</i> (test)
<i>Acceptance test</i> ¹				
Rank sum				
Appearance (color)	78 ^b	108 ^a	121 ^a	25.5**
Odor	81 ^b	108 ^a	117 ^a	16.0**
Taste	76 ^b	114 ^a	117 ^a	24.5**
Mouthfeel	73 ^b	116 ^a	124 ^a	63.0**
Overall impression	69 ^c	109 ^b	129 ^a	42.6***
<i>Preference test</i> ²				
Rank sum	67 ^c	105 ^b	134 ^a	44.3**
Most preferred (%)	8	24	69	
Preferred (%)	16	59	25	
Less preferred (%)	76	18	6	

Different superscript letters indicate significant ($*p<0.05$) or highly significant ($**p<0.01$) differences between the rank sums determined by Friedman test ($F=5.99$ or 9.21) and pairwise Least Significant Difference test

¹Higher rank sum indicate higher acceptance

²Higher rank sum indicate higher preference

press, and decanter on a 7-point hedonic scale ranging from ‘dislike very much’ (1) over ‘neither like nor dislike’ (4) to ‘like very much’ (7) evaluated by an untrained consumer panel ($n=51$)

horizontal filter press (108), and decanter (121) based on Friedman test ($p<0.05$ or $p<0.01$) presented in Table 3.

Similar observations were made for odor and taste. While the majority of the panel liked the decanter and horizontal filter pressed juices (odor: 86 and 71%, resp.; taste: 72 and 60%, resp., voting 5–7), approximately half of the panel disliked the spiral filter pressed juices [odor: 51% and taste: 48% votes for ‘dislike very much’ to ‘slightly dislike’ (1–3)]. Resultantly, the rank sums for spiral filter pressed juices were significantly lower (odor: 81 and taste: 76) compared to those of horizontal filter pressed and decanter made ones (odor: 108 and 117, resp.; taste: 114 and 117, resp.). The observations regarding odor and taste for the differently produced juices were partly unexpected, since descriptive sensory analyses did not indicate significantly different perceptions of characteristic blackcurrant attributes. Possibly, the odor- and taste-related ratings made by the untrained panelists of the consumer tests were influenced by a differing mouthfeel, originating from higher turbidity and cloud contents in the spiral filter-made juices as compared to the conventionally produced juices.

Table 4 Consumer acceptance and preference test of spiral filter press, horizontal filter press, and decanter made blackcurrant nectars evaluated by an untrained consumer panel ($n=51$)

	Spiral filter press	Horizontal filter press	Decanter	<i>F</i> (test)
<i>Acceptance test</i> ¹				
Rank sum				
Appearance (color)	76 ^c	105 ^b	126 ^a	29.9***
Odor	91 ^b	92 ^b	124 ^a	17.3**
Taste	98 ^a	95 ^a	114 ^a	5.2
Mouthfeel	99 ^{ab}	92 ^b	116 ^a	8.5*
Overall impression	77 ^b	106 ^a	124 ^a	26.0**
<i>Preference test</i> ²				
Rank sum	90 ^b	87 ^b	128 ^a	16.5**
Most preferred (%)	20	16	63	
Preferred (%)	37	39	25	
Less preferred (%)	43	45	12	

Different superscript letters indicate significant ($*p<0.05$) or highly significant ($**p<0.01$) differences between the rank sums determined by Friedman test ($F=5.99$ or 9.21) and pairwise Least Significant Difference test

¹Higher rank sum indicate higher acceptance

²Higher rank sum indicate higher preference

Here, the number of panelists disliking the spiral filter pressed juices' mouthfeel (higher viscosity and contents of solids as mentioned above) accounted for 50% compared to 24% scores between 5 and 7. In contrast, with regard to the decanter and horizontal filter press made juices, 76 and 74% panelists liked the mouthfeel, which was additionally confirmed by significantly higher rank sums (124 and 116) compared to the spiral filter pressed juice (rank sum: 73).

As indicated already by the single attributes, the overall impression of spiral filter pressed juices was comparably low, following a Gaussian distribution peaking at 'slightly dislike' (29%) and a majority of 53% of panelists disliking the overall impression compared to 82 and 71% liking the decanter and horizontal filter pressed juices overall impression confirmed also by significantly higher rank sums (129 and 109).

Blackcurrant nectars The results for the nectars made from the juices by diluting with water and adding sugar are shown in Fig. 5b. As already seen for juices, the appearance of nectars prepared from conventional pressing systems was higher rated (81 and 88% votes between 5 and 7) compared to that of the spiral filter press nectar (53% votes between 5 and 7). Even after dilution with 75% water, the visual differences described above may still affect consumers' acceptance. Widely similar rank sums for the attribute appearance

(color) were seen for the differently produced juices and the corresponding nectars (see Tables 3 and 4).

For odor and taste, a slightly different quantitative distribution was found for the spiral filter made products. While approximately half of consumers disliked the odor and taste of spiral filter pressed juices, for nectars, the highest share of votes was obtained at 6 (odor: 37%; taste: 33%) resulting in a total of 59% of consumers liking (5–7) the odor and 72% the taste of spiral filter press nectar.

As seen for juices, the likings of the nectars produced by horizontal filter press and decanter were high, with 63 and 82% of consumers liking the odor (hedonic score 5–7) and 65 and 81% liking the taste. Significantly highest odor rank sums were seen for decanter derived nectars (124) compared to similar rank sums in spiral and horizontal filter press derived ones (91–92).

By diluting and sweetening the juices, also the mouthfeel slightly changed in thereof derived nectars. While a low liking was seen for spiral filter pressed juices (50% of votes between 1 and 3), a peak was seen at 6 ('like') for nectars, with 35% of votes resulting in a general liking of 75% (votes between 5 and 7). 78 and 81% of consumers liked the mouthfeel of nectars produced from decanter and horizontal filter pressed juice, respectively.

Preparing nectars from spiral filter pressed juice clearly improved the overall impression as 72 and 28%, resp., of panelists voted between 5 and 7. The general liking for decanter and horizontal filter press made products was less affected when comparing juices to corresponding nectars (decanter: 82 and 88%, resp.; horizontal filter press: 71 and 68%, resp.). The acceptance test revealed significantly higher rank sums for the overall impression when produced from decanter (124) and horizontal filter pressed (106) juices compared to those made with the spiral filter press (77), see Table 4.

Consumer preference

Blackcurrant juices As already seen in the acceptance test, the preference for the spiral filter pressed juices was the lowest (Table 3), achieving a rank sum of 67, followed by that of the horizontal filter pressed (105) and decanter made juices (134). The share of panelists showing highest preference for decanter made juice accounted for high 69% compared to 24 and 8% for horizontal and spiral filter pressed juice, respectively. The proportion of panelists ranking the spiral filter pressed juice as 'least preferred' was considerably high (76%). It remains unclear, why no significant differences were seen for descriptive sensory analysis, but spiral filter pressed juices were rated significantly lower in the acceptance and preference tests. As men-

tioned above, the higher cloud content and turbidity of the spiral filter pressed juices had possibly affected the ratings of the untrained panelists of the consumer tests, while the trained panelists of the descriptive sensory tests might have been able to rate odor and taste more independently than the untrained panel. Another observation made by comparing the results obtained by HS-SPME–GC–MS to those received by acceptance and preference tests was that the spiral filter pressed juices with significantly highest amounts of total volatiles were significantly lower ranked in sensory tests. Vice versa, the decanter made juices with smallest concentrations of total volatiles were preferred. Whether or not these elevated concentrations of characteristic blackcurrant volatiles showing both pleasant and less pleasant aroma impressions (e.g., ‘cheesy-sweaty’ or ‘solvent-like’) had contributed to the disliking of the spiral filter made juices by the untrained panel cannot be deduced from our results.

Blackcurrant nectars The results of the preference tests of the blackcurrant nectars differed from those of the differently produced juices (Table 4). Whereas the significantly highest rank sum was still reached by decanter made nectars (rank sum: 128) with 63% of panelists showing the highest preference for those products, the rank sums and votes for ‘most preferred’ for horizontal and spiral filter pressed juices were similar (rank sums: 87 and 90; ‘most preferred’: 16 and 20%) and without showing statistically significant differences. One reason for this observation may be that the dilution and sweetening during nectar processing levels out the differences among the juices by, e.g., diluting less pleasant aroma compounds or masking effects due to the admixture of sugar.

Conclusions

As shown in this two-year study on semi-industrial scale, the selection of the dejuicing technology for processing blackcurrants was highly important for the concentrations of total volatiles in the juices. Highest levels of volatiles were found in spiral filter pressed juice, followed by horizontal filter press and decanter made samples. Moreover, also differing relative proportions of mono- and sesquiterpenes and oxygen-containing terpenoids but also esters were seen among the juices. However, contents of the blackcurrant characteristic trace constituent 4MMB, found by targeted analysis in similar amounts in all juices remained widely unaffected by the dejuicing system used. Whether the differing composition of volatiles in blackcurrant juices can be attributed to diminished losses due to oxygen exclusion during vacuum

dejuicing, their superior extraction during spiral filter press processing, or their enrichment in the suspended solids fraction may be subject matter of ongoing research.

Unexpectedly, descriptive sensory analysis did not reveal any significant differences between the three blackcurrant juices and derived nectars produced with the aforementioned three dejuicing technologies. However, the decanter made juices and nectars were clearly better accepted and preferred by untrained consumers, whereas those obtained by spiral filter press were clearly lower rated and those processed by horizontal filter press ranged intermediate. Noteworthy, further processing blackcurrant juices into nectars resulted in a better rating of the spiral filter pressed products that were then similarly rated as the horizontal filter pressed ones, however, still being outperformed by the decanter made nectars. The sensorial observations made for spiral filter derived products might be caused by consumers not being familiar with the higher cloud content of the product, also leading to an increased viscosity and a generally altered mouthfeel.

A limitation of our study was that the different pressing systems had not only led to different oxygen intake, but also have resulted in differing cloud contents and turbidity of the juices. As discussed herein, the cloud content may be relevant for aroma release and might influence the perception of other sensory attributes. Therefore, an experiment with differently pressed juices, but identical cloud content might be a goal of subsequent studies. Another limitation is that we had decided to conduct the enzymation of the fruit mash identically, i.e., without further protection against oxygen for all variants. It might be worth studying a spiral filter-pressed juice that had received further protection against oxygen during enzymation in order to elucidate the relevance of this processing step. Noteworthy, our analytical data clearly showed that there must have been a difference in oxygen intake due to the presence of much higher levels of an array of volatile compounds. Future studies may focus on yet unexplored aspects, such as assessing the impact of further processing steps like clarification and filtration on the sensory quality of clear spiral filter made berry juices and nectars.

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Data availability No datasets were generated or analysed during the current study.

Declarations

Conflict of interest In June 2024, a manufacturer of spiral filter presses (GEA, Düsseldorf, Germany) has declared to temporarily provide a new spiral filter press model to Geisenheim University in the first quarter of 2025 in the frame of a sponsoring agreement.

Compliance with ethical standards The study was explained to the consumers and the trained panelists prior to sensory evaluation. The products tested were safe for human consumption.

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