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ORIGINAL PAPER

HS–SPME–GC–MS profiling and sensory analyses of juices from red-fleshed 'Weirouge' apples made with innovative and conventional dejuicing systems

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Abstract

Volatiles, descriptive sensory profiles as well as consumer acceptance and preference of juices from red-fleshed 'Weirouge' apples produced in 2019 and 2020 with three different dejuicing systems were assessed. HS–SPME–GC–MS analyses revealed differences in the profiles of volatiles in juices processed in an oxygen-reduced atmosphere with an innovative spiral filter press as compared to those obtained using conventional systems, i.e., horizontal filter press and decanter. A total of 49 volatiles was tentatively assigned and permitted a clustering of the samples according to vintage and processing technology by multivariate statistics. Tentative markers to differentiate the individual samples were deduced from the multivariate models. In both years, each three 1,3-dioxanes and C_6 alcohols were revealed as discriminative markers of horizontal filter pressed juices. Descriptive sensory analysis by trained panelists revealed higher intensity scores of 'oxidized' and 'apple-like' orthonasal odors in juices produced by horizontal filter press and decanter as compared to those obtained by spiral filter press. The visual appearance of the spiral filter pressed juices was significantly higher rated compared to those obtained by conventional pressing systems as revealed by an untrained consumer panel ($n = 65$). In contrast, both odor and taste were lower rated, ultimately resulting in a clear-cut higher acceptance and preference of the decanter-made juices, followed by those obtained by horizontal and spiral filter press.

Keywords Apple juice · Red-fleshed apples · Spiral filter press · Volatiles · Sensory analysis · Oxidation

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
PC	Principal component
PCA	Principal component analysis
PLS-DA	Partial least squares discriminant analysis
SFP	Spiral filter press
VID	Variable identification coefficient

Introduction

Red-fleshed apples are popular due to their attractive color and high concentrations of potential health promoting (poly)phenols [1]. Apart from color and antioxidant properties, (poly)phenols contribute together with sugars and acids to aroma, taste, mouthfeel, and particularly astringency of fruits and derived juices [2]. In addition, the aroma is determined by volatiles generated from primary metabolites like fatty acids, amino acids or carbohydrates during ripening and postharvest storage as well as those altered during processing. Noteworthy, the human olfactory system merely recognizes a limited proportion of the numerous apple volatiles [3–5]. Potent aroma compounds of apples comprise *inter alia* hexanal, (*E*)-2-hexenal, 1-butanol, 1-hexanol, 1-octen-3-one, β-damascenone,

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dimethyl sulfide, ethyl butanoate, and ethyl 2-methylbutanoate [6]. Hereby, the aroma activity is determined by the concentration and odor threshold of the individual constituents [7].

Apple volatiles may be classified into aldehydes, alcohols, ketones, terpenes, and esters [4, 5]. The typical apple juice aroma is mainly determined by esters and aldehydes, accounting for ca. 80–90% of the total volatiles [8]. Some aldehydes like hexanal and (*E*)-2-hexenal, which are described as "green" or "grassy" [4, 9] are generated by chemical or enzymatic oxidation of linoleic and linolenic acid [10]. Thus, oxygen plays an important role in the genesis of such aldehydes and alcohols via lipoxygenase and subsequent lyase-mediated cleavage of the intermediate fatty acid hydroperoxides, often being compounds with six carbon atoms [11].

The most common dejuicing systems for apple juice production are decanter and horizontal filter press that are described in detail in our previous contribution and the respective literature [12, 13]. During conventional juice production, oxidation processes are unavoidable and occur immediately after fruit crushing [14]. The processing steps milling and extraction as well as subsequent steps like filtration and thermal treatment may result in losses or alterations of aroma compounds [15–17], affecting both analytical and sensory juice characteristics [18]. Oxygen has been found to be detrimental for color and oxidation-sensitive constituents, i.e., ascorbic acid, anthocyanins, and colorless (poly) phenols when processing red-fleshed apples into cloudy juices applying conventional dejuicing systems, namely a horizontal filter press and a decanter [12]. Our past work showed a better retention of color and the aforementioned constituents, when using an innovative spiral filter press. For instance, processing by spiral filter press resulted in a significantly higher retention of anthocyanins (47.89–74.91 mg/L) compared to 12.59–17.26 mg/L found in horizontal filter press or decanter-made juice. Moreover, ascorbic acid levels in the spiral filter pressed juices of 21.0–39.6 mg/L clearly exceeded those in the juices obtained by conventional dejuicing systems (4.5–10.7 mg/L). The latter system enables minimal input of oxygen during the entire process of juice production, as milling is conducted under nitrogen gas and the juice extraction cell is placed under reduced pressure. The resulting pressure gradient in the extraction cell causes a simultaneous de-aeration of the product and the juice to leave the extraction cell into an inert buffer tank. A detailed description and graphic illustration of the spiral filter system is shown elsewhere [12]. However, the impact of this promising processing technology on the volatiles and the sensory quality of apple juice has not been assessed in previous studies. In addition, merely a few studies have investigated volatiles and sensory characteristics of juices obtained from red-fleshed cultivars [19].

In the present study, cloudy juices from red-fleshed 'Weirouge' apples from 2 years (2019 and 2020) were produced at pilot plant scale (ca. 200 kg per batch) applying three dejuicing systems, i.e., hydraulic horizontal filter press, decanter, and spiral filter press. Their profiles of volatiles were characterized by HS–SPME–GC–MS analyses and multivariate statistics. Descriptive sensory analysis by trained panelists was conducted. Furthermore, consumer acceptance and preference of the juices obtained were assessed. A main focus of this work was to elucidate how spiral filter juice processing in an oxygen-reduced atmosphere influences the composition of volatiles and sensory characteristics of juices from red-fleshed apples.

Materials and methods

Production of cloudy juices from red-fleshed apples

The processing of red-fleshed apples (*Malus domestica* Borkh. cv. 'Weirouge') to cloudy juice has been described in detail by Wagner et al. [12]. The apples were purchased in both years from a commercial producer Bleichhof (Meckenheim, Germany). The apples of both vintages were harvested at full maturity as determined by the experienced producer and confirmed in our laboratory applying the starch-iodine test described by Sekse [20]. At the time of processing, the apples in 2019 were slightly more mature than those processed in 2020 as indicated by their softer flesh.

As described by Volz et al. [21], texture-loss occurs much faster in maturing red-fleshed apples as compared to white-fleshed varieties, resulting in a narrow time slot for processing. For this reason, and also due to refrained enzymatical treatment, the yields achieved and presented in our last work [12] of 30.3–35.3 and 35.3–70.1% in 2019 and 2020, respectively, were smaller than those commonly achieved in the industry.

In brief, 200 kg of red-fleshed apples for each one of two technical repetitions per year (2019 and 2020) were processed with three different pressing systems, namely a spiral filter press with an integrated mill (VaculiQ-1000, VaculiQ, Hamminkeln, Germany), a horizontal filter press (HPL 200, Bucher, Niederweningen, Switzerland), and a decanter (Z23-3, Flottweg, Vilsbiburg, Germany). Rotten or faulty apples were removed by hand prior to processing. In the spiral filter process, the apples were crushed with the integrated mill prior to pressing and the raw juice was collected in an inert atmosphere (N_2) buffer tank.

For conventional juice productions with horizontal filter press and decanter, the apples were crushed by a progressive cavity pump with an extended compression casing with an integrated cutting mechanism (open hopper pump BTM Seepex, Bottrop, Germany) and the raw juices were collected in a buffer tank without inert atmosphere according to conventional practice. Ascorbic acid was not added irrespective of the pressing system. For preservation, all juices were rapidly heated to ca. 78 °C with a fruit juice dispenser (PAS1-PS2-81-V2, Mabo, Eppingen, Germany). The juice for sensory evaluation was hot-filled into amber 0.75-L glass bottles and cooled back to 20 °C within ca 15 min. The temperature–time profile was recorded in our previous work and equal to a *P*-value of ca. 2.5 [12]. The samples for GC analyses were similarly hot-filled into 50-mL bottles and immediately frozen at − 20 °C.

HS–SPME–GC–MS analyses of apple juice volatiles

Volatiles were analyzed with a Trace GC, a DSQII quadrupole mass spectrometer, and a Triplus autosampler (Thermo Fisher, Dreieich, Germany) as reported previously including slight modifications [22]. Briefly, an aliquot of 1.0 mL of apple juice and 10 µL of the aqueous internal standard solution containing 0.05% (*v/v*) 2-methyl-1-pentanol (Sigma-Aldrich, Taufkirchen, Germany) were filled into a 10-mL headspace vial sealed with a PTFE-coated silicon rubber septum. After a pre-incubation for 5 min at 40 °C, the volatiles were isolated from the headspace for 40 min at the same temperature using a polydimethylsiloxane/divinylbenzene fiber (65 µm PDMS/DVB, Stable Flex®, Supelco 57293-U, Sigma-Aldrich, Dreieich, Germany). The sample was continuously mixed during the entire incubation period. After injection in the splitless mode for 2 min at 250 °C, the volatiles were separated on a fused silica capillary column coated with a polar polyethylene glycol stationary phase (30 m × 0.25 mm, film thickness $d_f = 0.25$ µm ZB-Wax, Phenomenex, Aschaffenburg, Germany). Carrier gas was helium at a constant flow rate of 1.2 mL/min, the modified temperature program was: isothermal hold at 40 °C (1 min), linear increase to 180 °C (5 °C/min), linear increase to the final temperature of 250 °C (10 °C/min) held isothermal for 5 min (total run time: 41 min). For validation of the peak identity, a slightly polar 5% phenyl 95% polydimethylsiloxane stationary phase was used (30 m × 0.25 mm, $d_f = 0.25$ µm ZB-5, Phenomenex), applying the same temperature program.

Electron impact (EI) mass spectra at 70 eV were recorded in the positive ion mode at a scan range of *m/z* 40–270 (scan frequency 5.4 Hz) between 0 and 15 min and *m/z* 40–300 (1.8 Hz) for the final segment. Linear retention indices (LRIs) determined on both columns were calculated according to van den Dool and Kratz [23] relative to *n*-alkanes (C7–C30 and C8–C20 for the ZB-Wax and the ZB-5 stationary phase, respectively). Individual volatiles were identified by comparing their mass spectra and LRIs to a commercial library (NIST mass spectral database, version Nist 05 Libraries for XCalibur (Thermo Fisher Scientific), NIST Chemistry WebBook [24], and literature data [25–27]. Concentrations were expressed µg 2-methyl-1-pentanol equivalents per 100 mL of juice. Odor qualities of the individual volatiles were not assessed experimentally but tentatively assigned on the basis of literature data [28–33].

Sensory evaluation

Sample preparation

Since the apples in 2020 were processed at the ideal ripening stage, juices from this vintage were used for descriptive sensory analysis as well as for consumers' acceptance and preference testing. Prior to sensory evaluation, the apple juices stored in amber glass bottles were brought to room temperature (ca. 90 min) and shaken before tasting, ensuring homogenic dispersion of cloud particles. For descriptive sensory analysis, aliquots of each ca. 50 mL were filled into 215 mL ISO wine tasting glasses of black color immediately prior to testing. For consumer acceptance and preference tests, 215 mL colorless and transparent ISO wine tasting glasses were used. Samples were encoded using random three-digit codes and presented in a randomized order.

Descriptive sensory analysis

The panel for descriptive sensory analysis consisted of 13 panellists (6 female, 7 male, aged between 20 and 62 years), regularly and specially trained for DLG (Deutsche Landwirtschafts-Gesellschaft) testing of juices and wines. The test was conducted in a sensory testing room according to DIN EN ISO 8589 [34] as described previously [35]. The attributes for orthonasal and retronasal evaluation were chosen by the panel in a first session by describing the sensory characteristics and a subsequent discussion guided by the panel leader. The six attributes 'fruity', 'green/grassy', 'oxidized', 'musty/mouldy', 'purity ("cleanness")' and 'apple-like' were chosen. For retronasal sensory evaluation, the same attributes were selected, in addition to the mouthfeel 'astringency'. The attributes were evaluated in a second session according to the panels' experience without providing reference samples. The intensities of the selected attributes were rated using a 9-point hedonic scale ranging from 'not detectable' (0) to 'very strong' (9).

Consumer acceptance test

The consumer acceptance test according to DIN 10974 [36] was performed as detailed elsewhere [35]. Briefly, a consumer panel ($n = 65$) recruited among staff and students at Geisenheim University (48 male, 17 female, aged 19–62 with a median at 27 years) was requested to rate the appearance (color), odor, taste, mouth feel, and overall impression on a 7-point hedonic scale ranging from ‘dislike very much’ (1) over ‘neither like nor dislike’ (4) to ‘like very much’ (7).

Consumer preference test

Consumer preference was assessed according to DIN 10974 [36] and DIN ISO 8587 [37]. The panel specified above was additionally asked to rank the three juices according to their personal preference (most preferred, mean preferred, and least preferred), prohibiting ties in rank order (forced choice) as previously reported [35].

Statistical analyses

In this work, the profile of volatiles and sensory attributes of differently produced red-fleshed apple juices were compared. Three pressing systems (spiral filter press, horizontal filter press, decanter) were used for juice productions of two technological replicates per year in 2019 and 2020 ($n = 2$ per year, $n = 4$ in total). Samples for analysis were taken on the day of production and stored at -20 °C prior to GC-analysis and at 4 °C prior to sensory evaluation. Analyses were conducted in duplicate using each two samples per pressing system (technological replicate) in 2019 and 2020. Concentrations of each analyte were presented as mean ± standard deviation. Analysis of variance (ANOVA) was performed ($\alpha = 0.05$) comparing concentrations of volatiles from the three pressing systems. If statistical significance was indicated by the ANOVA, a Tukey’s HSD test was conducted. ANOVA and Tuckey’s HSD test were calculated using JASP (version 0.16.3.—JASP Team, Amsterdam, The Netherlands, 2022).

The volatiles in the juices produced by three pressing systems in 2019 and 2020 were analyzed by unsupervised principal component analysis (PCA) and hierarchical cluster analysis (HCA) using Wards method of agglomeration and Euclidean distances to explore differences among the differently produced juices. In addition, tentative marker compounds were calculated from the absolute loadings and the variances explained by the PCs [38]. Subsequently, partial least squares discriminant analysis (PLS-DA) was calculated separately for juices produced in 2019 and 2020 using the concentration of volatiles as *X*-variables and the three different pressing systems as categorical *Y*-variables to reveal the particular impact of the processing technology on the volatiles. Most discriminative markers with absolute variable identification (VID) coefficients larger than 0.80 ($\text{VID} \geq |0.80|$) were deduced from the PLS-DA as previously reported [38]. Multivariate statistics were performed applying PLS toolbox version 9.1 in Matlab version R2021b (both MathWorks, Massachusetts, USA).

For evaluation of the consumer acceptance and preference, the hedonic scores were converted to ranks. A Friedman test, followed by pairwise least significant difference (LSD) test was conducted (DIN ISO 8587 [37]) for identification of significant ($p < 0.05$) and highly significant ($p < 0.01$) differences. Calculations were performed with Excel 2019 (Microsoft Corporation, Redmond, WA, USA).

Results and discussion

HS–SPME–GC–MS analyses of apple juice volatiles

The retention indices and concentrations of the 49 volatiles assigned in the juices produced with different pressing systems are shown in Table 1. The numerical largest substance group found were 19 esters followed by 11 alcohols. In addition, six aldehydes, three 1,3-dioxanes, two terpenes, and each one ketone, acid, norterpenoid, and phenylpropene were found. Each two substances (no. 17ab and 31ab) were not resolved on the ZB-Wax stationary phase used for quantitation.

The concentrations of total volatiles were higher in 2019 (ranging between 6009 and 6915 µg/100 mL; expressed as 2-methylpentan-1-ol equivalents), compared to those determined in the 2020 vintage. In the latter samples, the total amount was lower in the spiral filter pressed juice (3159 µg/100 mL) compared to the reference juices (horizontal filter press: 4814 µg/100 mL; decanter: 4505 µg/100 mL) as seen in Fig. 1. Within one vintage, horizontal filter pressed juices yielded highest concentrations of total volatiles, however, differences were found to be statistically insignificant at $p < 0.05$.

The group of esters was quantitatively the largest portion. In 2019, esters amounted to concentrations between 2150 and 2290 µg/100 mL, accounting for 31–38% of total volatiles in the differently produced juices after isolation by HS-SPME. In the 2020 produced juices, 1253–1688 µg/100 mL of esters were found, similarly accounting for 34–39% of total volatiles. The elevated concentrations of total esters in the slightly more mature apples of 2019 are in accordance with Flath et al. [39] and Kakiuchi et al. [40] who have reported increasing concentrations of esters with progressing fruit maturation.

After esters, alcohols are the second most important group of aroma contributing compounds in apples [41]. In our juices, they were the second largest numerical group and amounted to total concentrations of 815–1310 µg/100 mL in 2019 (Fig. 1) whereby horizontal filter pressed juices displayed significantly higher concentrations than those obtained by spiral filter press and decanter and 726–1404 µg/100 mL in 2020. In spiral filter pressed juices, alcohols accounted for 13 and 23% of the total volatiles in 2019 and 2020, respectively (reference juices: 15–19 and 24–29% in 2019 and 2020, resp.).

Table 1 HS–SPME–GC–MS analysis of volatiles in juices from red-fleshed apples obtained by spiral filter press (SFP), horizontal filter press (HFP), and decanter (Dec)

No.	Proposed structure	CAS	Odor quality	$LRI_{ZB\text{-}Wax}$		$LRI_{ZB\text{-}5}$		Concentration^{a} (2020)			Concentration^{a} (2019)			Class^{b}
				Exp.	Ref.	Exp.	Ref.	SFP	HFP	Dec	SFP	HFP	Dec	
1	Ethyl acetate	141-78-6	Solvent-like, fruity^{3}	884	884	< 800	< 800	10	30	25	63	65	40	Ester (ac)
2	Ethanol	64-17-5	Ethanol-like^{3}	933	933	< 800	< 800	2	10	5	8	11	7	Alcohol
3	2-Methylpropyl acetate	110-19-0	Fruity, apple, pear^{5}	1012	1012	< 800	< 800	5	6	7	13	13	12	Ester (ac)
4	Ethyl butanoate^{c}	105-54-4	Fruity^{3,4}	1032	1029	804	804	32	58	59	158	152	135	Ester
5	Ethyl 2-methylbutanoate^{c}	7452-79-1	Fruity^{3}	1046	1043	850	850	18	36	36	114	109	91	Ester
6	*n*-Butyl acetate	123-86-4	Red apple, banana^{5}	1068	1065	816	816	120	156	191	222	214	254	Ester (ac)
7	Hexanal^{c}	66-25-1	Tallowy, leaf-like^{3}, green, grassy^{4}	1077	1075	802	802	40	88	164	33	58	97	Aldehyde
8	2-Methylbutyl acetate	624-41-9	Apple, fruit^{5}	1116	1116	881	877	229	269	358	301	255	300	Ester
9	1-Butanol^{c}	71-36-3	Fruity^{3}, malty, solvent-like^{4}	1148	1148	< 800	< 800	28	43	49	46	46	52	Alcohol
10	*n*-Pentyl acetate	628-63-7	Apple, fruity, banana^{5}	1167	1167	918	912	30	36	55	46	32	46	Ester (ac)
11	2-Methylbutanol	137-32-6	Malty^{3,4}, solvent-like^{4}	1209	1209	< 800	< 800	47	78	72	45	43	39	Alcohol
12	(*E*)-2-Hexenal^{c}	6728-26-3	Apple-like^{3}, green apple-like, bitter, almond-like^{4}	1215	1215	852	850	74	178	253	178	182	204	Aldehyde
13	Butyl 2-methylbutanoate	15,706-73-7	Apple, fruity^{5}	1225	1226	1044	1044	17	11	12	46	29	49	Ester
14	Ethyl hexanoate	123-66-0	Fruity^{3}	1228	1228	1001	1001	44	53	48	74	62	69	Ester
15	Ethyl (*E*)-2-butenoate (ethyl tiglate)	5837-78-5	Fruity, caramel^{7}	1232	1232	947	949	5	15	11	13	10	12	Ester
16	3-Methyl-2-butenyl acetate (prenyl acetate)	1191-16-8	Green apple, banana^{7}	1245	1250	928	929	41	28	33	19	7	9	Ester (ac)
17ab	3-Octanone + 1-Pentanol	106-68-3 71-41-0	Herb, butter, resin^{6} Fusel-like, mild^{5}	1249	1248 1252	n.d. n.d.	– –	3	3	3	11	8	5	Ketone* Alcohol*
18	*n*-Hexyl acetate	142-92-7	Fruity^{3}	1269	1267	1016	1011	408	448	546	691	547	790	Ester (ac)
19	Octanal	124-13-0	Fatty^{3}	1285	1285	n.d.	–	19	16	19	8	4	4	Aldehyde
STD	2-Methylpentan-1-ol (IS)	105–30-6	–	1303	1303	–	–	410	410	410	410	410	410	-
20	(*Z*)-2-Heptenal	57,266-86-1	Unknown	1320	1319	n.d.	–	18	10	13	13	7	4	Aldehyde
21	(*Z*)-3-Hexenyl acetate	3681-71-8	Sweet^{3}, green, banana-like^{4}	1325	1324	n.d.	–	27	25	35	28	15	24	Ester (ac)
22	(*E*)-2-Hexenyl acetate	2497-18-9	Pleasant fruity^{7}	1331	1330	1018	997	61	237	80	57	203	75	Ester (ac)
23	6-Methyl-5-hepten-2-one	110-93-0	Pepper, mushroom, rubber^{5}	1334	1333	1000	986	67	37	55	281	216	245	Ketone*
24	1-Hexanol^{c}	111-27-3	Green, flowery^{3}	1356	1356	871	874	379	831	646	404	812	523	Alcohol
25	(*Z*)-3-Hexen-1-ol	928-96-1	Leaf-like^{3}, lettuce-like^{4}	1385	1385	n.d.	–	1	5	4	1	6	3	Alcohol
26	Nonanal	124-19-6	Tallowy, fruity^{3}, citrus-like, soapy^{4}	1388	1388	1105	1101	41	57	44	9	6	6	Aldehyde
27	(*E*)-2-Hexen-1-ol	928-95-0	Green fruit, caramel^{5}	1405	1406	867	867	75	281	134	54	173	66	Alcohol
28	Butyl hexanoate	626-82-4	Grass, green apple^{5}	1410	1409	1188	1186	39	42	32	57	64	67	Ester
29	Hexyl butanoate	2639-63-6	Green^{5}	1412	1413	1188	1188	69	47	57	124	65	80	Ester
30	2-Methyl-4-pentyl-1,3-dioxane (DS 1)$^{\#}$	–	Unknown	1418	1418^{1}	1170	–	84	396	286	415	892	597	Dioxane

Table 1 (continued)

No.	Proposed structure	CAS	Odor quality	LRI$_{ZB\text{-}Wax}$		LRI$_{ZB\text{-}5}$		Concentration[a] (2020)			Concentration[a] (2019)			Class[b]
				Exp.	Ref.	Exp.	Ref.	SFP	HFP	Dec	SFP	HFP	Dec	
31ab	Hexyl 2-methylbutanoate +(*E*)-2-Octenal	10,032–15-2 2548-87-0	Apple, grape[5] Green, nut, fat[6]	1421	1425 1425	1236 1062	1237 1062	106	74	96	199	98	107	Ester
32	1-Octen-3-ol	3391-86-4	Mushroom[6]	1451	1451	992	992	6	5	3	4	5	5	Alcohol
33	1-Heptanol	111-70-6 53,535-33-4	Woody, heavy, oily, faint, aromatic, fatty[7]	1455	1455	981	976	20	14	16	11	10	9	Alcohol
34	6-Methyl-5-hepten-2-ol	1569-60-4	Flowery, green, honey- mushroom-like[8]	1464	1464	1007	1003	19	15	14	43	35	32	Alcohol
35	2-Methyl-4-(2′(*Z*)-pentenyl)-1,3-dioxane[#]	–	Unknown	1480	1475[1]	1177	–	33	173	95	129	288	166	Dioxane
36	2-Methyl-4-pentyl-1,3-dioxane (DS 2)[#]	202,188-43-0	Unknown	1491	1485[1]	1212	–	2	16	8	17	102	34	Dioxane
37	Decanal	112-31-2	Orange skin-like, flowery[3]	1494	1494	n.d.	–	21	10	6	3	2	1	Aldehyde
38	1-Octanol	111-87-5	Fresh, orange-rose[7]	1557	1557	n.d.	–	51	31	44	45	47	49	Alcohol
39	Hexyl hexanoate	6378-65-0	Apple, cucumber[5]	1602	1603	1382	1385	59	69	51	107	138	102	Ester
40	Butyl octanoate	589-75-3	Fruit[6]	1606	1604	n.d.	–	9	7	4	20	36	26	Ester
41	(*Z*)-5-Octen-1-ol	64,275-73-6	Green, fatty[7]	1616	1616	n.d.	–	98	92	101	153	121	118	Alcohol
42	Estragole	140-67-0	Liquorice-like[3]	1666	1666	1200	1200	56	39	54	41	30	27	Benzenoid*
43	2-Methylbutanoic acid	116-53-0	Sweaty, sweet[3]	1695	1700	n.d.	–	17	11	11	31	35	21	Acid
44	(*Z*,*E*)-α-Farnesene	26,560-14-5	Unknown	1718	1715	1497	1484	24	25	17	185	76	70	Terpene
45	(*E*,*E*)-α-Farnesene	502-61-4	Wood, sweet[6]	1744	1740	1511	1509	532	573	543	1520	1388	1189	Terpene
46	(*E*)-β-Damascenone[c]	23,696-85-7	Honey-like, fruity, sweet[3], baked apple-like, grape, juice-like[4]	1811	1819	1382	1382	42	69	61	41	63	53	Norterpene*
47	Ethyl 5-(*Z*)-3-hydroxyoctenoate[#]	–	Unknown	1944	1941[2]	1327	–	30	60	48	138	132	96	Ester

Volatiles were tentatively assigned based on mass spectra and linear retention indices (LRI) on a polar (ZB-Wax) and a slightly polar (ZB-5) stationary phase. Compounds labelled by[#] were not contained in the mass spectral library used for compound identification and assigned on the basis of literature data

Dec decanter, *DS* diastereomers, *HFP* horizontal filter press, *IS* internal standard, *n.d.* not detected, *SFP* spiral filter press

Unless otherwise indicated, LRI reference values were taken from the NIST webbook [24]. Further references: [1]Dietrich et al. [25], [2]Beuerle et al. [26]. Odor quality: [3]Rychlik et al. [28], [4]Czerny et al. [29], [5]Espino-Diaz et al. [30], [6]Acree and Arn [31], [7]Burdock and Fenaroli (2010) [32], [8]Werkhoff et al. [33]

[a] Means ($n = 2$) were expressed in µg 2-methyl-1-pentanol equivalents (IS) per 100 mL of apple juice determined using a polar (ZB-Wax) stationary phase

[b] Volatiles were classified into alcohols, aldehydes, dioxanes, esters (including acetates, ac), terpenes, and not resolved/others (acids, ketones, norterpenoids, and phenylpropenoids) whereby the latter category is labelled by asterisks

[c] Volatiles proposed by Steinhaus et al. as most potent odorants of juices from conventional, white-fleshed apples (cv. ‘Golden Delicious’)

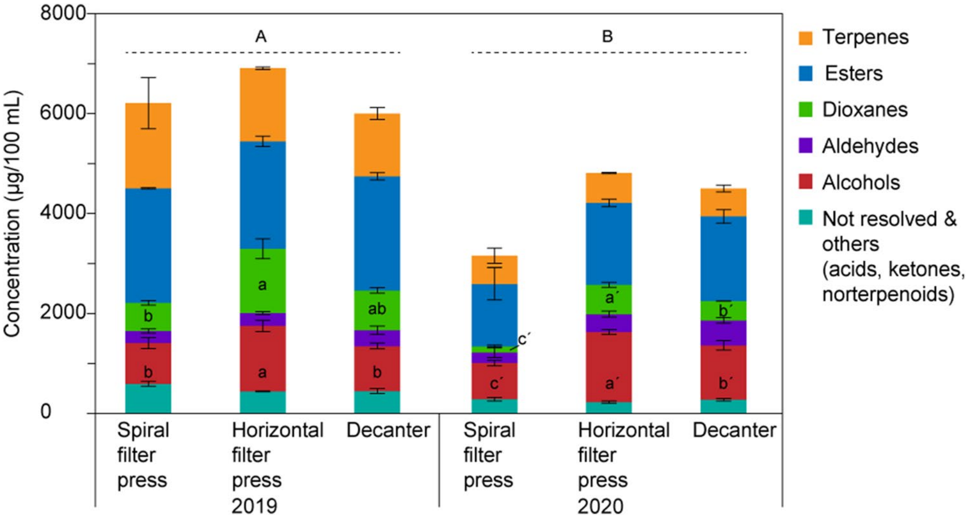

Fig. 1 Total concentrations of volatiles (as 2-methyl-1-pentanol equivalents in µg/100 mL) categorized in the main classes in juices from red-fleshed apples obtained by spiral filter press, horizontal filter press, and decanter. Significant differences of means ($p < 0.05$) within one vintage are indicated by lower case letters and those of the total volatiles between the 2019 and 2020 samples by capital letters

In the group of aldehydes, the amounts in the reference juices in 2019 and 2020 (horizontal filter press: 258 and 359 µg/100 mL; decanter: 316 and 500 µg/100 mL, resp.) were slightly but not significantly higher compared to the spiral filter pressed juices (244 and 213 µg/100 mL, resp.). Aldehydes accounted for 4–5 and 7–11% of total volatiles in 2019 and 2020, respectively. In homogenized fruit tissues and juices, elevated concentrations of aldehydes like hexanal and hexenals are found [42, 43].

The proportions of the individual compound classes found herein were within the ranges reported in literature for 47 monovarietal cloudy apple juices including 6 red-fleshed varieties, even though a different SPME fiber was used for isolation of volatiles [8]. In the aforementioned study, esters accounted for 35–65% of total volatiles in juices from red-fleshed varieties with larger proportions of alcohols and aldehydes compared to those in the remaining juices. Among all samples, alcohols and aldehydes accounted for 8–56 and 3–53% of the volatiles [8].

The method established in this work allowed the detection of seven out of nine volatiles that have been proposed as the most potent odorants of apple juice by Steinhaus et al. [6]. Noteworthy, Steinhaus et al. [6] studied common apples (cv. 'Golden Delicious'), while we investigated red-fleshed apples. In descending order according to their odor activity values in unprocessed juice [6], the most important aroma compounds according to Steinhaus et al. [6] are (*E*)-β-damascenone (no. 46), hexanal (7), ethyl 2-methylbutanoate (5), 1-octen-3-one (not found), (*E*)-2-hexenal (12), dimethyl sulphide (not found), ethyl butanoate (4), 1-hexanol (24), and 1-butanol (9). In 2019, differences in the contents of those mentioned key volatiles in our juices were found to be statistically insignificant, except for 1-hexanol (green, flowery) that was found in elevated concentrations in horizontal filter pressed juice compared to those obtained by spiral filter press and decanter (Table 1). In 2020, significantly lower concentrations of (*E*)-2-hexenal (apple-like, almond-like) and 1-hexanol were found in the spiral filter pressed juices compared to the reference samples. Moreover, levels of 1-butanol (fruity, malty, solvent-like) in the decanter-made juices significantly exceeded those in the spiral filter pressed juices. The possible contribution of the key volatile (*E*)-β-damascenone is discussed in section "Supervised discrimination by PLS-DA".

Aldehydes, alcohols, and esters are *inter alia* derived from fatty acids by β-oxidation or lipoxygenase pathway [44, 45]. As β-oxidation occurs in intact fruit [46, 47], lipoxygenase reactions seem more relevant for juice processing after tissue disruption [48]. The ambient air during horizontal filter press and decanter juice processing and the higher oxygen amounts found in the juices [12] may result in elevated concentrations of lipoxygenase-derived volatiles during the processing steps prior to pasteurization, particularly of C_6 aldehydes, C_6 alcohols, and their esters (see also "Supervised discrimination by PLS-DA" section and Table 1). Some of these C_6 alcohols and C_6 aldehydes, like some of the key aroma compounds mentioned above, have been reported as being responsible for the green leaf- and apple-like aroma found in conventional apple juice [14, 19, 49].

Unsupervised classification by PCA and HCA

For deeper understanding of the differences among the juices resulting from processing technology, a principal component analysis (PCA) was calculated on the basis of the concentrations of individual volatiles. Figure 2 shows the scores and loadings of the first two principal components (PCs), which together described 61.4% of the variance among the data set. On PC 1 (43.3%), the samples were separated by the production year, 2019 and 2020. On PC 2 (18.0%), the driving factor for the sample separation was the processing

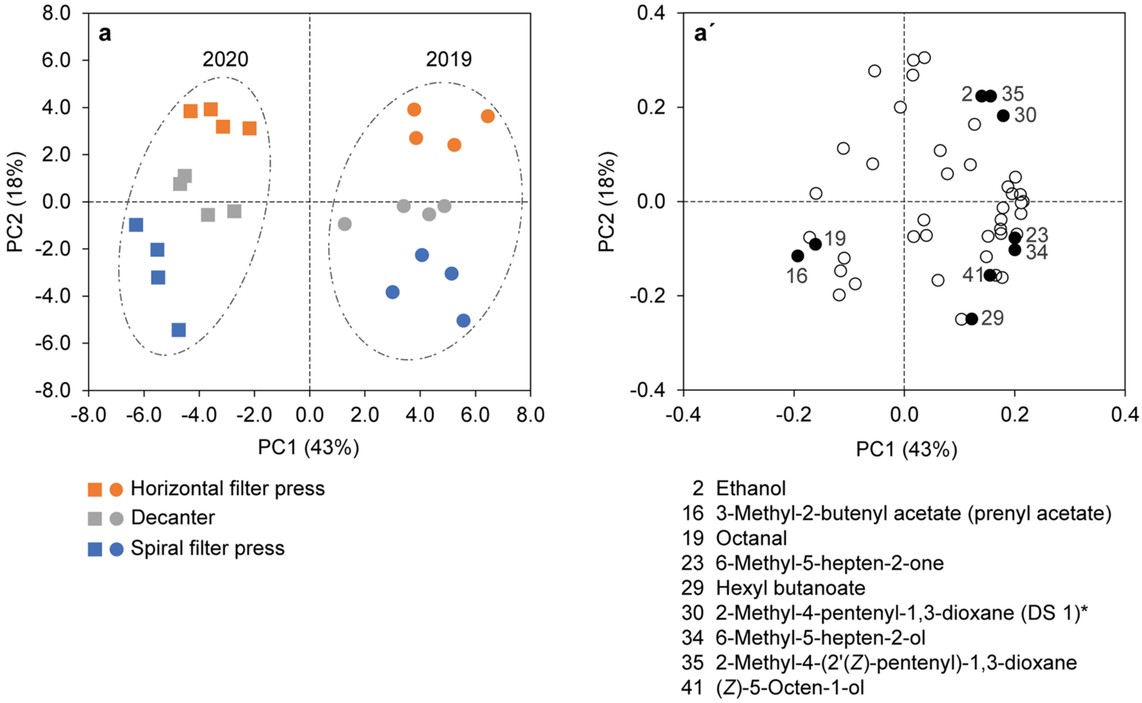

Fig. 2 PCA scores (**a**) and loadings plot (**a´**) calculated on the basis of all volatiles determined in juices from red-fleshed apples obtained by spiral filter press, horizontal filter press, and decanter. The ellipses in the scores plot illustrate clusters from hierarchical cluster analysis (HCA). Tentative marker compounds calculated by PCA are indicated by filled circles (calculations, see Supplementary Information)

technology. These observations were also confirmed by the HCA (cf. circles in Fig. 2), that grouped the samples by year of production.

The volatiles with the strongest impact on separating the samples are labelled in the PCA loadings plot in Fig. 2a´. The corresponding loadings on PC1 and PC2 are compiled in Table S1 provided as Online Supplementary Information. These volatiles displayed both, negative and positive loading on PC 1, which means that marker compounds are found for both years of processing. Hereby, two markers were positively correlated to the 2020´s samples, namely 3-methyl-2-butenyl acetate (16) and octanal (19). The higher concentration of 3-methyl-2-butenyl acetate may be related to the raw material processed at an optimal stage of maturity in 2020, compared to slightly overripe apples in 2019, since the concentrations of esters have been reported to decrease after harvest due to enzymatic reactions or their release to the environment [50–52]. Noteworthy, the slightly higher ethanol (2) levels in the juices processed in 2019 may indicate that the apples of this vintage may already have reached senescence.

In 2019, one subgroup with volatiles linked to the horizontal filter pressed juice was found. The volatiles 2-methyl-4-pentenyl-1,3-dioxane (DS 1) (30), 2-methyl-4-(2′(*Z*)-pentenyl)-1,3-dioxane (35), and ethanol (2), with positive loadings on PC 2 contributed to the clustering and separation of the juices produced by horizontal filter press. Their occurrence in horizontal filter pressed juices may be linked to the operating principles of the horizontal filter press. While the spiral filter press and decanter are continuous pressing systems, the horizontal filter press works in a batchwise mode. As described in Wagner et al. [12], an initial amount of 40 kg apple mash was filled in the pressing chamber and 20 kg mash was added every 2 min after every press cycle duration, consisting of a pressing process, followed by loosening up the pomace in the pressing chamber, where ambient air is soaked into the press. The oxygen exposition during the entire dejuicing process, which lasted 60 min, is more intense as compared to that of fast and continuous pressing systems like a decanter. During this extended period, enzymatic reactions after disintegration of the fruit tissue, e.g., generation of carbonyls and possibly also hydrolysis of glycoside-bound 1,3-diols such as 3-hydroxyoctyl-β-D-glucoside [53] may ultimately result in elevated concentrations of 1,3-dioxanes.

The volatiles hexyl butanoate (29) and (*Z*)-5-octen-1-ol (41) were found in higher quantities in spiral filter pressed juices.

The marker volatiles 6-methyl-5-hepten-2-one and 6-methyl-5-hepten-2-ol are degradation products of α-farnesene (45), i.e., the prevailing terpene found in our apple juices (Table 1), and were linked to the decanter-made juices. 6-Methyl-5-hepten-2-one has been reported to be an oxidation product of α-farnesene [54, 55], also being generated during storage of intact apples in an oxygen-containing atmosphere within the first 2 month [56]. In our juices, particularly those processed in 2019, α-farnesene may be oxidized, resulting in 6-methyl-5-hepten-2-one that may be reduced to the corresponding alcohol 6-methyl-5-hepten-2-ol in an enzyme-catalyzed reaction. This would agree with the elevated α-farnesene levels of 1520 µg/100 mL in the spiral filter pressed juices compared to the 1388 and 1189 µg/100 mL found in the horizontal filter and decanter-made juices, respectively, in 2019 (see Table 1). Noteworthy, α-farnesene levels in the juices of 2020 merely ranged between 532 and 573 µg/100 mL, which again may indicate that they were obtained from slightly less mature apples. In agreement with our observations, elevated levels of the 6-methyl-5-hepten-2-ol (34) have been previously reported in apples harvested at a later stage, i.e., at a more progressed maturity [57].

Supervised discrimination by PLS-DA

To further explore the particular impact of the three processing technologies on the volatiles, a separate partial least squares discriminant analysis (PLS-DA) was calculated for each year (2019 and 2020) and employed as discriminative variable selection method. Figure 3 shows the resulting scores and loadings plots for the first two latent variables (LVs).

In 2020, LV1 and LV2 explained 85% of the *Y*-variance (Fig. 3a and a´) and together accounted for 90% of explained

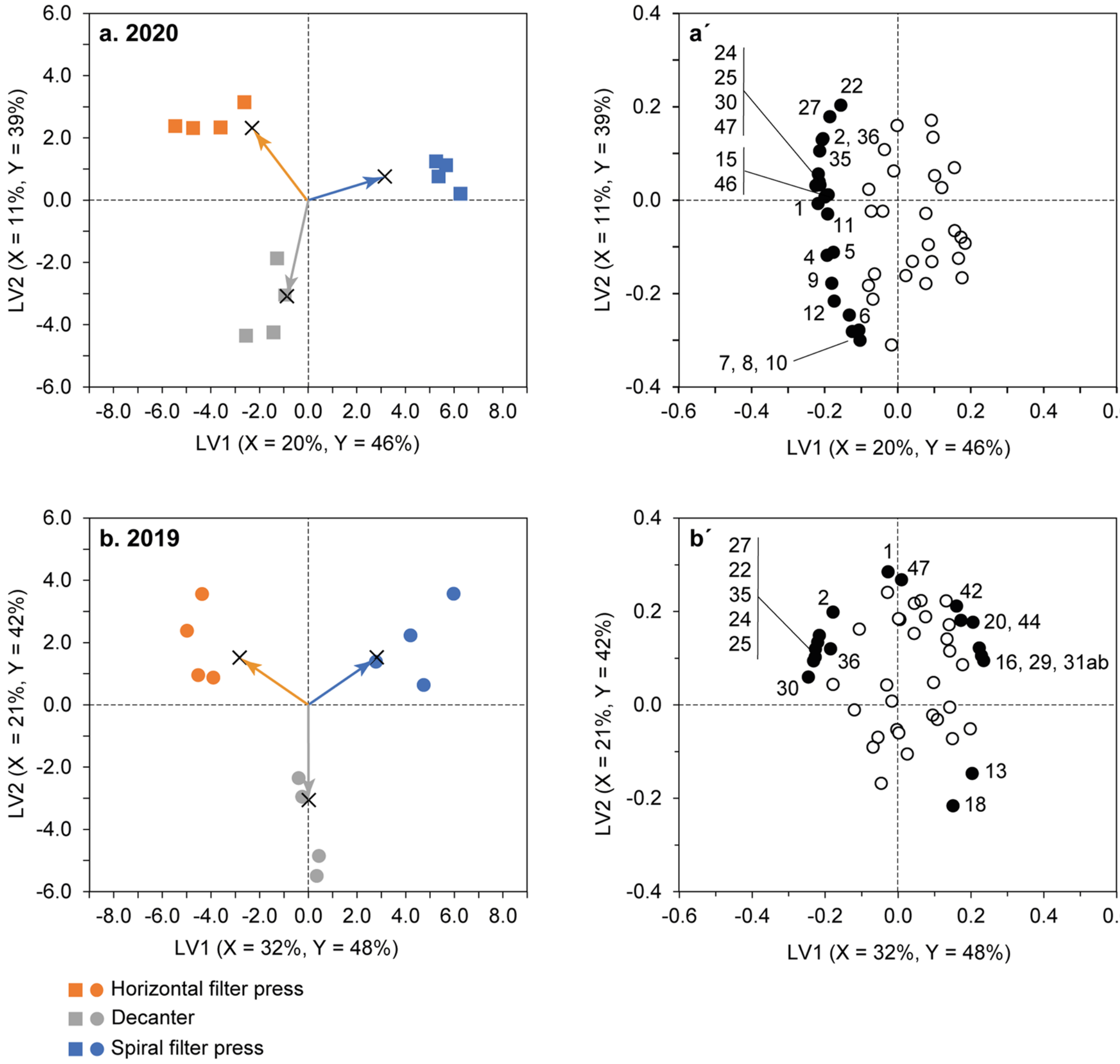

Fig. 3 PLS-DA scores (left) and loadings plot (right) calculated on the basis of the volatiles in juices from red-fleshed apples obtained by spiral filter press, horizontal filter press, and decanter in 2020 (**a**, **a´**) and 2019 (**b**, **b´**). Arrows indicate the correlation loadings for the categorical *Y*-variables, i.e., the processing technology. Tentative marker compounds (VID ≥ |0.80|) illustrated by black-filled circles are compiled in Table 2, their assignment and concentrations in Table 1

variance in 2019 (Fig. 3b and b´). The groups typifying the different pressing systems were clearly divided into three separate clusters. Most discriminative volatiles were determined by calculating variable identification coefficients (VID) and compounds with VID ≥|0.80| are summarized in Table 2.

In 2020, all discriminative volatiles of the spiral filter pressed juice showed negative VIDs, indicating smaller concentrations of these markers in the spiral filter pressed juices compared to those found in the samples obtained by horizontal filter press and decanter. In 2019, six compounds were positively correlated to the spiral filter pressed juices, mainly comprising unsaturated volatiles such as, e.g., (*Z*,*E*)-farnesene (44) or 3-methyl-2-butenyl acetate (16) that may be prone to oxidative degradation during processing involving oxygen. Hereby, the latter volatile has been reported to exert green apple- and banana-like odors (Table 1).

In the horizontal filter pressed juices, compounds with positive VIDs comprised the identical eight volatiles for juices processed in 2020 and 2019, respectively. The discriminative compound ethyl 5-(*Z*)-3-hydroxyoctenoate (47) was additionally found as a marker in 2020. Discriminative volatiles, irrespective of the vintage, were ethanol (2), the three C_6 alcohols 1-hexanol (24, green, flowery),

Table 2 Discriminative volatiles (VID ≥|0.80|) separating apple juices produced by spiral filter press, horizontal filter press, and decanter

Year	Spiral filter press		Horizontal filter press		Decanter	
2020	VID	Identity (peak no.)	VID	Identity (peak no.)	VID	Identity (peak no.)
	− 0.81	Ethyl 2-methylbutanoate (5)[a]	0.96	Ethanol (2)	0.92	*n*-Pentyl acetate (10)
	− 0.83	Ethyl (*E*)-2-butenoate (15)	0.95	(*E*)-2-Hexen-1-ol (27)	0.91	Hexanal (7)[a]
	− 0.84	2-Methyl-4-pentenyl-1,3-dioxane (DS 2) (36)	0.95	2-Methyl-4-pentenyl-1,3-dioxane (DS 2) (36)	0.87	2-Methylbutyl acetate (8)
	− 0.84	(*E*)-2-Hexenal (12)[a]	0.94	2-Methyl-4-(2′(*Z*)-pentenyl)-1,3-dioxane (35)	0.85	(*E*)-2-Hexenal (12)[a]
	− 0.84	1-Butanol (9)[a]	0.89	1-Hexanol (24)[a]	0.84	*n*-Butyl acetate (6)
	− 0.84	Ethanol (2)	0.89	(*Z*)-3-Hexen-1-ol (25)		
	− 0.84	2-Methylbutanol (11)	0.88	2-Methyl-4-pentenyl-1,3-dioxane (DS 1) (30)		
	− 0.87	(*E*)-β-Damascenone (46)[a]	0.87	(*E*)-2-Hexenyl acetate (22)		
	− 0.89	2-Methyl-4-(2′(*Z*)-pentenyl)-1,3-dioxane (35)	0.84	Ethyl 5-(*Z*)-3-hydroxyoctenoate (47)		
	− 0.89	Ethyl butanoate (4)[a]	− 0.82	3-Methyl-2-butenyl acetate (16)		
	− 0.91	Ethyl 5-(*Z*)-3-hydroxyoctenoate (47)	− 0.90	1-Octanol (38)		
	− 0.92	1-Hexanol (24)[a]				
	− 0.94	(*Z*)-3-Hexen-1-ol (25)				
	− 0.96	Ethyl acetate (1)				
	− 0.96	2-Methyl-4-pentenyl-1,3-dioxane (DS 1) (30)				
2019	0.95	(*Z*,*E*)-α-Farnesene (44)	0.97	(*Z*)-3-Hexen-1-ol (25)	− 0.84	Ethyl 5-(*Z*)-3-hydroxyoctenoate (47)
	0.95	Hexyl butanoate (29)	0.95	2-Methyl-4-(2′(*Z*)-pentenyl)-1,3-dioxane (35)	− 0.90	Ethyl acetate (1)
	0.94	Hexyl 2-methylbutanoate + (*E*)-2-Octenal (31 ab)	0.95	(*E*)-2-Hexen-1-ol (27)		
	0.93	3-Methyl-2-butenyl acetate (16)	0.95	(*E*)-2-Hexenyl acetate (22)		
	0.84	Estragole (42)	0.95	2-Methyl-4-pentenyl-1,3-dioxane (DS 1) (30)		
	0.82	(*Z*)-2-Heptenal (20)	0.93	1-Hexanol (24)[a]		
			0.89	Ethanol (2)		
			0.80	2-Methyl-4-pentenyl-1,3-dioxane (DS 2) (36)		
			− 0.80	*n*-Hexyl acetate (18)		
			− 0.90	Butyl 2-methylbutanoate (13)		

DS diastereomers, *VID* variable identification coefficient

[a] Volatiles proposed by Steinhaus et al. as most potent odorants of juices from conventional, white-fleshed apples (cv. ‘Golden Delicious’)

(*Z*)-3-hexen-1-ol (25, leaf- and lettuce-like), and (*E*)-2-hexen-1-ol (27, green fruit, caramel) and a derived ester, i.e., (*E*)-2-hexenyl acetate (22, pleasant fruity). In addition to these markers mainly deriving from lipoxygenase reactions, all three 1,3-dioxanes found in the apple juices, i.e., two diastereomers of 2-methyl-4-pentenyl-1,3-dioxane (30 and 36) and their unsaturated analogue 2-methyl-4-(2′(*Z*)-pentenyl)-1,3-dioxane (35) were positively correlated to the horizontal filter pressed juices of both vintages (cf. PCA discussed in "Supervised discrimination by PLS-DA" section).

In the group of samples processed by decanter, marker compounds with positive VIDs were merely found in the 2020 juices, namely the C_6 aldehydes hexanal (7, tallowy, leaf-like green) and (*E*)-2-hexenal (12, apple- and almond-like, bitter), and the acetate esters *n*-butyl acetate (6, red apple, banana), 2-methylbutyl acetate (8, apple, fruit), and *n*-pentyl acetate (10, apple, fruity, banana), whereas no positively correlated volatiles were found in 2019.

Owing to their importance to the flavor, the five C_6 aldehydes and alcohols (i.e., hexanal (7), (*E*)-2-hexenal (12), 1-hexanol (24), (*Z*)-3-hexen-1-ol (25), and (*E*)-2-hexen-1-ol (27)) that were all found among the discriminative markers deduced by PLS-DA (see Table 2) were considered separately (data not shown). In the spiral filter pressed juices, significantly lower concentrations of the aforementioned C_6 aldehydes and alcohols were found in 2020 (569 µg/100 mL) compared to those in the reference juices obtained by horizontal filter press (1383 µg/100 mL) and decanter (1202 µg/100 mL). In 2019, the concentrations in the spiral filter pressed juice (671 µg/100 mL) were significantly lower compared to the horizontal filter pressed sample (1232 µg/100 mL) and slightly but not significantly lower compared to the decanter-made juice (892 µg/100 mL). Most likely, lower oxygen levels in the spiral filter pressed juices and the shorter exposure duration as compared to that of the horizontal filter pressed juices may have resulted in lower concentrations of these C_6 compounds, being important contributors to the characteristic apple odor and flavor [41].

Another discriminative marker negatively correlated with spiral filter pressed juices in 2020 was (*E*)-β-damascenone (46) (Table 2). This important aroma compound with a very low perception threshold has been reported to exert flavors of stewed or baked apples, honey, and sweetness to apple juices [7]. (*E*)-β-Damascenone was found in lower concentrations (41 and 42 µg/100 mL in 2019 and 2020, resp.) in the spiral filter pressed juices compared to those obtained by horizontal filter press and decanter (53–69 µg/100 mL). Noteworthy, (*E*)-β-damascenone has been reported to be generated by co-oxidation of carotenoids involving different oxidases such as lipoxygenase (LOX) and polyphenoloxidase (PPO) [58].

All three 1,3-dioxanes found, i.e., two diastereomeric 2-methyl-4-pentyl-1,3-dioxanes (30 and 36) and methyl-4-(2′(*Z*)-pentenyl)-1,3-dioxane (35), were among the discriminative volatiles. Total concentrations of dioxanes in 2019's spiral filter pressed juice (561 µg/100 mL, Fig. 1) were slightly but not significantly lower compared to the decanter samples (798 µg/100 mL), but significantly lower compared to those of the horizontal filter pressed samples (1283 µg/100 mL). In 2020, significant differences in total dioxanes, which have been proposed as important aroma compounds of cider, were found between the three pressing systems, with lowest levels in the spiral filter juice (119 µg/100 mL), followed by decanter-made samples (389 µg/100 mL) and the horizontal filter pressed juice (585 µg/100 mL).

The slightly more advanced maturity in 2019 may possibly have resulted in higher concentrations of 1,3-dioxanes in this vintage. Different mechanisms for the genesis of 1,3-diols and derived 1,3-dioxanes in apples have been discussed in literature [26]. 1,3-Diols may represent intermediates of the fatty acid metabolism, possibly involving catabolic β-oxidation or lipoxygenase reaction [59] and anabolic de novo synthesis [53]. Beuerle and Schwab [60] have investigated the biosynthesis of (*R*)-1,3-diols in stored apples during β-oxidation of linoleic acid. As a stereoselective step, enoyl-CoA hydratase catalyzes the hydroxylation of 2-(*Z*)-octenyl-SCoA, resulting in (*R*)-3-hydroxyoctanoyl-SCoA. The latter may be converted into a corresponding ester or the reduction of the (*R*)-3-hydroxy octanoic acid intermediate may result in (*R*)-1,3-octanediol. In an analogous pathway, linolenic acid was transformed into its unsaturated analogue (*R*)-5(*Z*)-octene-1,3-diol [60].

In apples, those 1,3-diols, which are accumulated in free or glycosylated form [26, 27, 53], may react with apple or fermentation derived carbonyls like acetaldehyde to form cyclic dioxanes [10]. Therefore, such dioxanes have mainly been described in apple cider [25, 61], but also in pear fruit [62]. In addition, the release of glycoside-bound 1,3-diols, e.g., by enzymatic or acid hydrolysis, may also be a crucial factor determining the genesis of such 1,3-dioxanes [10].

The origin of acetaldehyde in our apple juices that might have triggered the formation of 1,3-dioxanes remains somehow obscure. Acetaldehyde has been reported to derive from oxidation of ascorbic acid [63] and the oxidation of ethanol in a coupled autoxidation reaction of phenolic compounds in wine [64]. Assuming similar pathways, the high oxygen exposure in the horizontal filter press in conjunction with the comparatively long period until thermal enzyme inactivation may have resulted in elevated acetaldehyde and 1,3-diol levels and thus, the boosted formation of derived 1,3-dioxanes. Kavvadias et al. [61] have reported additional dioxanes in apple wine, presumably deriving from the reaction of aldehydes other than acetaldehyde with the aforementioned 1,3-diols such as propanal, butanal, 2-methylpropanal, hexanal, 3-methylbutanal, and 2-methylbutanal as well as the ketones acetone and 2-butanone. In particular, cyclic 1,3-dioxanes have been reported to contribute to cider aroma [10, 65]. The conversion of unpleasant aldehydes and ketones into dioxanes may have a positive impact on aroma, as the dioxanes exert a weak but pleasant "green note" flavor [61].

In our juices, the concentration of saturated 1,3-dioxanes (30 and 36) was higher compared to that of the unsaturated form (35), thus being in accordance with the literature as linoleic acid concentrations in apples exceed those of linolenic acid [10]. Whereas only insignificant differences between the levels of (poly)phenols, color, and genuine ascorbic acid in juices from the decanter and the horizontal filter press were observed in our previous work [12], these juices were clearly separated based on their volatiles by PCA and PLS-DA. Here, in particular, the prolonged oxygen exposure during the non-continuous horizontal filter pressing compared to the continuous decanter dejuicing may have resulted in a differing composition of volatiles.

Descriptive sensory analysis

Similar odor profiles of the three juices were seen for both, orthonasal and retronasal evaluation (see Supplementary Information Table S2 and Supplementary Information Figure S3). In all juices, medium to high scores were reached for both, orthonasal and retronasal ratings of 'fruity' (intensity score 4.85 to 5.85) and 'purity' (intensity score 5.15 to 5.62), with low musty/mouldy impressions (intensity score 1.85 to 3.08). The orthonasal attribute 'oxidized' was significantly higher rated by trend ($p < 0.1$) in the horizontal filter pressed juices (intensity score 3.62) compared to the juices derived by decanter and spiral filter press (intensity scores 2.28 and 2.15, resp.). Regarding the operation principle of the horizontal filter press explained in "Unsupervised classification by PCA and HCA" section, this observation seems reasonable, as reactions with the oxygen of ambient air might have resulted in the generation of aroma compounds perceived as 'oxidized'. Significant ($p < 0.05$) differences were seen for the orthonasal attribute 'apple-like', where the spiral filter pressed juice (intensity score 4.15) was rated slightly lower compared to the horizontal filter pressed sample (intensity score 5.31) and significantly lower compared to the decanter (intensity score 6.15) made juice. This is in accordance to the analytical data. The total amounts of volatiles, whose odor quality has previously been described as 'apple' (volatiles no. 3, 6, 8, 10, 12, 13, 16, 28, 31, 39, and 46 in Table 1), were significantly lower in the spiral filter pressed juice (657 µg/100 mL) compared to the decanter-made juice (1054 µg/100 mL) and slightly but not significantly lower compared to the juice produced with the horizontal filter press (866 µg/100 mL). Three of these volatiles were found as PLS-DA marker compounds in the juice made with the decanter in 2020, namely *n*-butyl-acetate (6), 2-methylbutyl acetate (8), and *n*-pentyl acetate (10). One apple-like marker (3-methyl-2-butenyl acetate (16)) was found to be discriminative for the horizontal filter pressed juice.

Consumer acceptance and preference

Consumer acceptance

The frequency distributions of the consumer acceptance test are presented in Fig. 4, the corresponding rank sums are compiled in Table 3. The visual appearance was rated the highest in the spiral filter produced juices, where 74% of the panel voted a score of 6–7. Merely 14% rated the appearance below 4. The acceptance of the reference juices (horizontal filter press and decanter) was in general lower rated. The distribution in the decanter juice

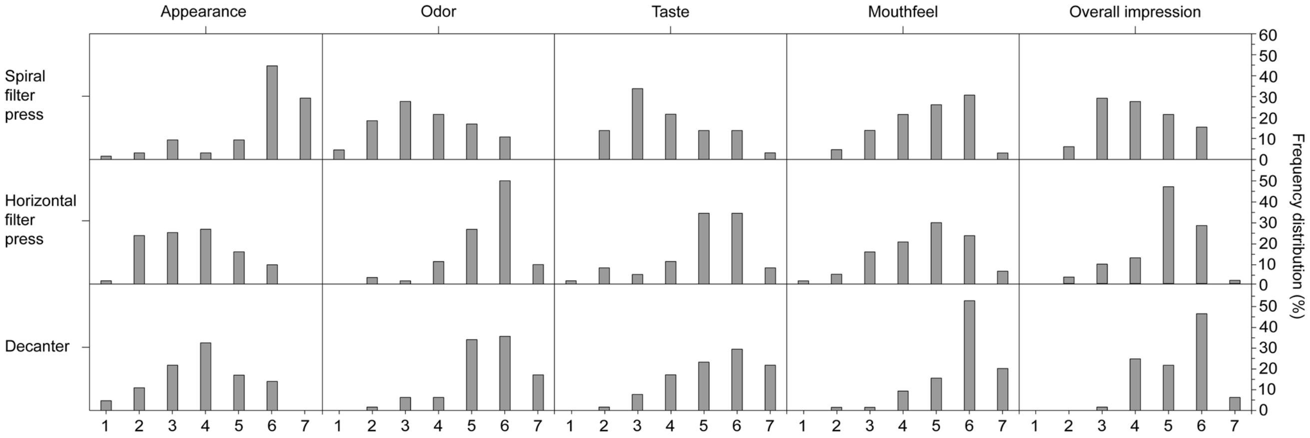

Fig. 4 Frequency distribution of hedonic scores of appearance (color), odor, taste, mouthfeel, and overall impression of juices obtained from red-fleshed apples by spiral filter press, horizontal filter press, and decanter on a 7-point hedonic scale ranging from 'dislike very much' (1) over 'neither like nor dislike' (4) to 'like very much' (7) evaluated by a consumer panel ($n = 65$)

Table 3 Consumer acceptance and preference test of apple juices produced with spiral filter press, horizontal filter press, and decanter assessed by a consumer panel ($n = 65$)

	Spiral filter press	Horizontal filter press	Decanter	F (test)
Acceptance test[1]				
Rank sum				
Appearance	171^a	105^b	115^b	41.5**
Odor	83^b	154^a	154^a	56.9**
Taste	94^b	141^a	156^a	38.1**
Mouthfeel	115^b	112^b	164^a	32.2**
Overall impression	99^c	134^b	158^a	31.5*
Preference test[2]				
Rank sum	107^c	130^b	153^a	16.3*
Most preferred (%)	14	31	55	
Preferred (%)	37	38	25	
Least preferred (%)	49	31	20	

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

[1] Higher rank sums indicate higher acceptance

[2] Higher rank sums indicate higher preference

approximated a Gaussian distribution, peaking at ‘neither like or dislike’ (4) with 32% of all ratings. The acceptance for the horizontal filter pressed juice was lower, merely 24% rated those juices higher than 4, the majority (74%) rated the appearance between 2 and 4. Based on Friedman and LSD analyses, the spiral filter pressed juice had a highly significant ($p < 0.01$) higher liking score regarding appearance (Table 3). The consumer acceptance for the appearance agreed with our previous research, which showed significantly higher redness (CIE-a* of 30.5) and lower brownish hues (h° of 44.1) of the fresh spiral filter pressed compared to the reference juices (horizontal filter press: a* 11.4, h° 63.0. Decanter: a* 13.3, h° 64.9) [12]. This intensely bright reddish color seemed to be appreciated by the consumers. The slightly better rated appearance of the decanter compared to that of the horizontal filter made juice may also be related to its slightly higher a* value.

The odor of the reference juices (horizontal filter press and decanter) was scored higher than 4 by 84–86%, and the spiral filter pressed juice merely by 28% of the panelists. The odor acceptance of the spiral filter pressed juice approximated a Gaussian distribution, peaking at 3 (‘slightly dislike’). A similar frequency distribution was revealed for the taste, with a higher acceptance for the reference juices (74–76% higher than 4) compared to the spiral filter pressed juice (31%). Resultantly, highly significant differences in the rank sums of odor and taste were found between the reference juices and the spiral filter juice (Table 3).

Interestingly, the mouthfeel of the decanter-made juice was rated high 6–7 by 72% of the consumers, resulting in a significantly higher rank sum (164) as compared to that of the spiral and horizontal filter pressed juices (115 and 112, resp.). This may be attributed to the smallest cloud content and viscosity in decanter-made juices as reported previously [12].

The overall impression showed a clear and significant ($p < 0.05$) customer preference for the decanter-made juices (rank sum 158), followed by that of the horizontal (134) and spiral filter pressed samples (99). Even though the percentage of people that liked the odor, taste and texture very much (7 points) was 17–22%, merely 6% of the panel rated the overall impression of the decanter-made juice to 7 points.

Consumer preference

The results from the consumer acceptance were clearly confirmed by the preference test (Table 4). The decanter-made juice achieved the highest rank sum of 153, followed by those obtained by horizontal and spiral filter press of 130 and 107, respectively (significant differences at $p < 0.05$). Accordingly, 55% of the panelists ranked the decanter-made sample ‘most preferred’. Less panelists preferred the horizontal filter and spiral filter pressed juices (31 and 14%, resp.). As described in section "HS–SPME–GC–MS analyses of apple juice volatiles", higher concentrations of C_6 compounds were found in the juices produced by horizontal filter press and decanter compared to those obtained by spiral filter press. Although, the total amounts in the 2020’s juices used for sensory analysis were similar in the decanter and horizontal filter made juice (652 µg/100 mL), a clear-cut preference of the decanter-made juice was revealed. As described in section "Supervised discrimination by PLS-DA" this may be attributed to certain aldehydes and esters that were found to be characteristic to the decanter juices in 2020 (Table 2) that are linked with fruity, banana- and apple-like aromas [30]. The odor of the PCA marker 6-methyl-5-hepten-2-one (23) has been described to exert grassy, fresh and green-fruity [66] but also pepper-, mushroom-, and rubber-like odors (see Table 1). These pleasant aroma compounds may substantially contribute to the high overall acceptance and preference of the decanter-made juices. The impact of the individual volatiles on the sensory properties of differently produced apple juices may be subject matter of ongoing research.

Conclusions

The present study revealed a substantial impact of the processing technology on the composition of volatiles and the results of sensory tests regarding cloudy juices made from red-fleshed apples, for which ascorbic acid is usually not added due to the contained labile anthocyanins. The HS–SPME–GC–MS method applied herein permitted the detection of 49 different volatiles. Their total concentrations in juices processed in 2019 exceeded those of the samples obtained from slightly less mature apples in 2020. PCA and HCA calculated on the basis of the concentrations of the individual volatiles permitted the clustering of the samples according to the year of processing. PLS-DA clearly separated groups typifying the three pressing systems and, moreover, permitted to deduce most discriminative volatiles to differentiate the juices. The analytical and statistical workflow reported herein may be highly instrumental for continuative studies assessing the impact of different processing technologies on fruit juice volatiles. Interestingly, consumers clearly preferred the decanter-made juices, followed by those obtained by horizontal and spiral filter press. As revealed in this study, the bright intense red color of spiral filter pressed juices was highly appreciated by the consumers. Such visual product perceptions may largely influence the buying decision of beverages, but also the advertisement provided on the label, e.g., for gentle processing methods. Our past work showed that spiral filter pressed juices are indeed also richer in potentially health beneficial compounds like ascorbic acid and (poly)phenols including anthocyanins [12] as chemical and enzymatic oxidation reactions are reduced. However, besides from a better retention of functional constituents working under exclusion of oxygen regarding odor, taste, and ultimately the overall impression, a certain degree of oxidation may be crucial for genesis of the typical apple juice aroma. Descriptive analysis also revealed not just higher intensity scores of 'oxidized' but also 'apple-like' odors in juices produced by horizontal filter press and decanter under oxygen exposure, which agreed with the HS–SPME–GC–MS data. This shows, that tentative assumptions regarding the odor characteristics may be drawn based on the analytical data obtained, even though a semiquantitative approach was applied. It is also worth mentioning that consumers are commonly not familiar with the distinct aroma of spiral filter pressed juices, which may have an impact on their acceptance and preference. Moreover, spiral filter pressed juices may not only be marketed as such, but provide interesting ingredients for innovative mixed beverages with pleasant color hues containing high levels of antioxidants.

The contribution of 1,3-dioxanes to the aroma of apple juice as well as their genesis merits further investigation. Continuative studies may additionally target at optimization of the aroma characteristics of the spiral filter-pressed apple juice while concomitantly permitting the retention of functional constituents.

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Data availability Data will be made available upon request.

Declarations

Conflict of interest The authors declare no conflict of interest.

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

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