



Impact of defined thermomechanical treatment on the structure and content of dietary fiber and the stability and bioaccessibility of polyphenols of chokeberry (*Aronia melanocarpa*) pomace



Vera Schmid^{a,b}, Jan Steck^c, Esther Mayer-Miebach^{a,*}, Diana Behnlian^a, Karlis Briviba^d, Mirko Bunzel^c, Heike P. Karbstein^b, M. Azad Emin^b

^a Max Rubner-Institut (MRI), Department of Food Technology and Bioprocess Engineering, Karlsruhe, Germany

^b Institute of Process Engineering in Life Sciences, Section I: Food Process Engineering, Karlsruhe Institute of Technology, Karlsruhe, Germany

^c Institute of Applied Biosciences, Department of Food Chemistry and Phytochemistry, Karlsruhe Institute of Technology, Karlsruhe, Germany

^d Max Rubner-Institut (MRI), Department of Physiology and Biochemistry of Nutrition, Karlsruhe, Germany

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ABSTRACT

Dietary fiber is a potential replacement for other ingredients such as starch in reformulated extruded breakfast cereals. Analysis of chokeberry pomace powder revealed a total dietary fiber content of 57.8 ± 2 g/100 g with 76% being insoluble, 20% high molecular soluble and 4% low molecular soluble dietary fiber. The fiber polysaccharide composition was analyzed in detail by using a variety of analytical approaches. Extrusion-like processing conditions were studied in a Closed Cavity Rheometer enabling the application of defined thermal (temperature range 100–160 °C) and mechanical treatments (shear rates between 0.1 s^{-1} and 50 s^{-1}) to chokeberry pomace powder. Application of temperatures up to 140 °C irrespective of the mechanical treatment does not remarkably alter dietary fiber structure or content, but reduces the initial content of total polyphenols by about 40% to a final content of 3.3 ± 0.5 g/100 g including 0.63 ± 0.1 g/100 g of anthocyanins, 0.18 ± 0.02 g/100 g of phenolic acids and 0.090 ± 0.007 g/100 g of flavonols, respectively. The retained polyphenols are fully bioaccessible after *in vitro* digestion, and antioxidant capacity remains unchanged as compared to the untreated pomace powder. Glucose bioaccessibility remains unaffected, whereas glucose content is reduced. It is concluded that chokeberry pomace powder is a good source of dietary fiber with the potential to partially substitute starch in extruded breakfast cereals.

1. Introduction

Crispy breakfast cereals often contain large amounts of easy to digest carbohydrates, e.g. starches, which are rapidly degraded after consumption resulting in high blood glucose levels. The consumption of such high glycaemic food is associated with an increased risk for metabolic disorders, such as obesity, type 2 diabetes, or cardiovascular diseases. Dietary fiber (DF) has been suggested as a potential starch replacer to be used in the reformulation of cereal products towards healthier food (Clemens et al., 2012; FDA, 2018a,b; Garcia-Amezquita, Tejada-Ortigoza, Serna-Saldivar, & Welte-Chanes, 2018). It mainly consists of non-protein plant cell wall polymers and is neither digested

in nor absorbed from the small intestine.

Breakfast cereals are produced by High-Temperature Short-Time extrusion processing exposing ingredients to thermomechanical stress. This may induce changes in the structures of DF polysaccharides, thereby affecting their physiological properties including their impact on the glycaemic response (Brümmer, Meuser, Van Lengerich, & Niemann, 2002). Insoluble dietary fiber (IDF) is supposed to be solubilized resulting in larger amounts of high molecular weight soluble dietary fiber (HMW-SDF) (Dust et al., 2004; Sobota, Sykut-Domańska, & Rzedzicki, 2010). However, comprehensive knowledge on the stability of DF and its potentially altered impact on the glucose release from products in which starch has been partially replaced is still needed

Abbreviations: AOX, antioxidant capacity; CCR, closed cavity rheometer; CPP, chokeberry pomace powder; DF, dietary fiber; TDF, total dietary fiber; IDF, insoluble dietary fiber; HMW-SDF, high molecular weight soluble dietary fiber; HPAEC-PAD, high performance anion-exchange chromatography with pulsed amperometric detection; LMW-SDF, low molecular weight soluble dietary fiber; NSP, non-starch polysaccharides; PMAA, partially methylated alditol acetates; PP, polyphenols; TPP, total polyphenols

* Corresponding author.

E-mail address: esther.mayer-miebach@mri.bund.de (E. Mayer-Miebach).

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(Leonard, Zhang, Ying, & Fang, 2020).

Chokeberry (*Aronia melanocarpa*) pomace, a residue from juice production is rich in DF and bioactive secondary plant metabolites, e.g. polyphenols (PP) (Kulling & Rawel, 2008). The cell wall constituents and associated PP are enriched within the pomace whereas water soluble PP as well as glucose, fructose and sorbitol are mostly extracted with the juice (Mayer-Miebach, Adamiuk, & Behnlian, 2012; Sójka, Kołodziejczyk, & Milala, 2013).

The cell wall of chokeberry pomace typically consists of cellulose, hemicelluloses and large amounts of pectins (Nawirska & Kwaśniewska, 2005). These groups of polysaccharides contain different structural elements, potentially being differently resistant to thermomechanical stress. To the best of our knowledge, there are neither studies characterizing cell wall polysaccharides of chokeberry pomace in detail nor their modifications due to thermomechanical stress. PP are generally sensitive to oxygen, light, pH-changes and temperature, and PP losses during extrusion processing are reported (Camire, Dougherty, & Briggs, 2007; Hirth, Preiß, Mayer-Miebach, & Schuchmann, 2015; Leonard et al., 2020; White, Howard, & Prior, 2010). However, both improved PP bioaccessibility and bioavailability, as well as enhanced antioxidant capacity (AOX) resulting from thermal processing have also been described (Liu et al., 2019; Marinelli, Padalino, Conte, Del Nobile, & Briviba, 2018).

The inclusion of fruit pomace in extruded products has been the target of numerous studies and reviews (Leonard et al., 2020; Quiles, Campbell, Struck, Rohm, & Hernando, 2018; Wang, Gu, & Ganjyal, 2019). Yet it is hardly possible to predict the effect of processing parameters on the retention of bioactive compounds, as in extrusion processing, thermal and mechanical treatment are linked to each other and their respective impact on the target compounds is hard to determine (Emin & Schuchmann, 2017). Furthermore, DF and starch interactions due to extrusion processing cannot be excluded (Quiles et al., 2018).

Aiming at the reduction of the amount of easily digestible carbohydrates in extruded cereal products, our overall goal is the evaluation of chokeberry pomace fiber as a partial replacer of starch. The objective of this study was to gain insights into the impact of extrusion-like thermal and mechanical treatment on bioactive constituents as basis for a target-oriented design of extrusion processes. For this, we used a Closed Cavity Rheometer (CCR) for the treatment of chokeberry pomace powder (CCP). The CCR, in contrast to extrusion processing, enables to apply defined thermal and mechanical stresses either combined or separately. Evaluated were the DF structure, the DF, PP and sugar contents of a commercial chokeberry pomace powder (CPP) and the effect of treatment on DF and PP stability. Furthermore, after *in vitro* digestion of CPP and treated pomace material, glucose release, bioaccessibility of PP and AOX were estimated.

2. Materials and methods

2.1. Materials and reagents

Commercial chokeberry pomace powder (Aronia Original Naturprodukte GmbH, Germany) was used. All chemicals and reagents were of analytical purity grade and purchased from Merck KGaA (Germany), unless otherwise stated. Amyloglucosidase (E-AMGDFPD, from *Aspergillus niger*, 36,000 U/g), α -amylase (E-PANAA, from pig pancreas 100,000 U/g), protease (E-BSPRPD from *Bacillus licheniformis* 9000 U/g), Celite 545 and *endo*-arabinanase (EC 3.2.1.99, from *A. niger*, 9 U/mg) were obtained from Megazyme (Ireland), Amberlite FPA53 and Ambersep 200 were from Rohm and Haas Europe (Germany). Thermostable α -amylase (Thermamyl 120 L, EC 3.2.1.1, from *B. licheniformis*, 120 KNU/g), protease (Alcalase 2.5 L, EC 3.4.21.62, from *B. licheniformis*, 2.5 AU/g), amyloglucosidase (AMG 300 L, EC 3.2.1.3, from *A. niger*, 300 AGU/g) were a gift from Novozymes (Denmark). PP standards cyanidin-3-O-glucoside ($\geq 96\%$), cyanidin chloride ($\geq 97\%$),

5-caffeoylquinic acid (TLC), quercetin-3-O-glucoside ($\geq 99\%$), quercetin dihydrate ($\geq 99\%$) as well as Rotihydroquant C5 and Rotihydroquant D used for Karl Fischer titration were purchased from Carl Roth GmbH & Co. KG (Germany). Ultrapure water was used throughout all experiments.

2.2. Thermomechanical treatment

A Closed Cavity Rheometer (CCR; RPA Elite, TA Instruments, USA), which enables to vary temperature (T), shear rate ($\dot{\gamma}$) and treatment time (t) independently (Koch, Hummel, Schuchmann, & Emin, 2017), was used for defined thermal and/or mechanical treatment.

CPP was first adjusted to water contents (c_w) of approx. 12% or 22% using a Thermomix (Vorwerk, Germany) for about 1 min and stored at 8 °C for at least 72 h to ensure uniform hydration (pre-treated material). Approximately 5 g were filled into the cavity of the measurement cell, which was then sealed under a pressure of 4.5 MPa to avoid water evaporation at elevated temperatures.

In order to identify treatment temperatures resulting in potential structural modifications of CPP polymers preliminary studies were performed, that is, the rheological behavior of pre-treated materials was analyzed in temperature sweep tests (Supporting information, Fig. S1).

Based on these preliminary data the study design was scheduled: treatment parameters and analyses performed are given in Table 1. Samples C, D, E were used to evaluate mechanical stress; B, E and H to evaluate thermal stress and short-time treatment; G and I thermal stress and long-time treatment; E and F water content of the pre-treated material; sample A was used to evaluate a treatment temperature without effect on *in vitro* glucose release. Each thermomechanical treatment was carried out twice. After treatment the samples were packaged in vacuum bags, stored at -80 °C, and analyzed as described.

2.3. Dietary fiber analysis

2.3.1. Dietary fiber isolation

IDF and HMW-SDF fractions of CPP and CCR treated materials were preparatively isolated as described by Bunzel, Ralph, Marita, Hatfield, and Steinhart (2001). In brief, 1 g of mortar ground powder was subsequently incubated with thermostable α -amylase, protease and amyloglucosidase. IDF was recovered as residue and HMW-SDF was precipitated after addition of four volumes of ethanol to the supernatant. Both fractions were washed and dried as described in Bunzel et al. (2001) and used without correction for residual protein and ash content.

2.3.2. Monosaccharide constituents

The monomer composition of IDF and HMW-SDF polysaccharides was determined after sulphuric acid hydrolysis (Saeman, Bubl, & Harris, 1945) and methanolysis (DeRuiter, Schols, Voragen, & Rombouts, 1992), respectively. Hydrolyzed samples were analyzed by high performance anion-exchange chromatography with pulsed amperometric detection (HPAEC-PAD) on a Dionex ICS-5000 system equipped with a CarboPac PA-20 column (6.5 μ m, 150 \times 3 mm, ThermoFisher Scientific, Germany) as described by Wefers and Bunzel (2015).

2.3.3. Monosaccharide linkage patterns

Linkage analysis was performed according to Gniechwitz, Reichardt, Blaut, Steinhart, and Bunzel (2007) and Wefers and Bunzel (2015). Briefly, both DF fractions were swollen in dimethyl sulfoxide, and polysaccharides were methylated twice (using NaOH and methyl iodide), extracted into dichloromethane and subsequently hydrolyzed using trifluoroacetic acid. Partially methylated monosaccharides were reduced by adding sodium borodeuteride in aqueous ammonia and subsequently acetylated by adding acetic anhydride and 1-

Table 1

Treatment parameters and analyses performed. T: temperature (°C), $\dot{\gamma}$: shear rate (s^{-1}), t: time (min), c_w : water content (g/100 g), DF: dietary fiber, PP: total polyphenols, AOX: antioxidant activity, CPP: chokeberry pomace powder.

Sample name	Treatment parameters				Analysis					
	T	$\dot{\gamma}$	t	c_w	DF		PP		Glucose Release	AOX
					Content	Structure	Content	Bioaccessibility		
CPP	–	–	–	3.5 ± 0.6	A	A	A	A	A	A
Pre-treated	–	–	–	–	A	NA ^a	A	NA	NA	NA
A	80 ^b	50	1	12	NA	NA ^a	NA	A	A	A
B	100	50	1	12	A	NA ^a	A	NA	NA	NA
C	140	0.1	1	12	A	NA ^a	A	NA	NA	NA
D	140	12.5	1	12	A	NA ^a	A	NA	NA	NA
E	140	50	1	12	A	A	A	A	A	A
F	140	50	1	22	A	A	A	NA	NA	NA
G	140	50	20	12	A	A	A	NA	NA	NA
H	160	50	1	12	A	A	A	A	A	A
I	160	50	20	12	A	A	A	A	A	A

A: analyzed, NA: not analyzed.

^a DF structure modification not to be expected according to temperature sweep analysis.

^b Thermal treatment below 100 °C with no expected effect on *in vitro* glucose release.

methylimidazole. Resulting partially methylated alditol acetates (PMAA) were analyzed by GC-MS and GC-FID using molar response factors as described by Sweet, Shapiro, and Albersheim (1975).

2.3.4. Oligosaccharide screening

Pectic arabinan profiling was performed by liberating arabinan oligosaccharides from both IDF and HMW-SDF using an *endo*-arabinanase as described by Wefers and Bunzel (2016). Relative quantification was performed via HPAEC-PAD equipped with a CarboPac PA-200 column (5.5 μ m, 250 × 3 mm, ThermoFisher Scientific, Germany) using raffinose as internal standard and previously published relative response factors (Wefers & Bunzel, 2016).

2.3.5. Total dietary fiber content

Two aliquots (250 mg) of each CPP, pre-treated and CCR treated materials (ground by mortar and pestle) were analyzed for their IDF, HMW-SDF, low molecular weight soluble dietary fiber (LMW-SDF) and total DF (TDF) contents according to AOAC Official Method 2011.25 (AOAC, 2012) as modified by McCleary, Sloane, and Draga (2015). LMW-SDF was analyzed by HPLC-RID (1100 series, Agilent Technologies, Germany) using two size exclusion columns (Toyo TSKgel, G2500PWXL, 7.8 × 300 mm, Tosoh, Germany) in series (McCleary et al., 2015).

2.4. In vitro digestion

In vitro digestion of CPP and CCR treated materials (50 mg each) was performed according to Gille, Trautmann, Posten, and Briviba (2016) with some modifications. Briefly, to simulate the gastric phase, porcine pepsin in 0.1 N HCl was added to achieve the final concentration of 1875 U/mL, and the pH was adjusted to 2.2–2.4, followed by shaking at 37 °C in a water bath for 1 h. Subsequently, the intestinal phase was initiated by adding porcine bile acid (9 mM), pancreatin (85 U/mL trypsin activity), and α -amylase (400 U/mL). The pH was adjusted to 7.2–7.6, followed by a treatment with nitrogen gas and shaking at 37 °C in a water bath for 2 h. After digestion, samples were collected and glucose release and PP bioaccessibility were analyzed.

2.5. Sugar, sugar alcohol analysis

Glucose, fructose, sucrose and sorbitol contents of LMW-SDF fractions, prepared according to the procedure described in Section 2.3.5 (AOAC digests), from CPP, pre-treated material and CCR treated CPP were analyzed using test kits (R-Biopharm AG, Germany).

In order to quantify glucose after *in vitro* digestion, 0.5 mL of sample (Section 2.4.) was added to 1 mL of ethanol and centrifuged (17,000g, 20 °C, 10 min). The supernatant was filtered (MWCO 3 kDa, Carl Roth GmbH, Germany), and the filtrate was incubated with amyloglucosidase (2.7 U/mL) for 1 h at 37 °C. Following filtration (MWCO 3 kDa, Carl Roth GmbH, Germany) samples were analyzed on an HPLC system (LC 626 System, Waters Corporation, Milford, MA) equipped with a 717Plus autosampler. A SUGAR KS-801 (300 × 8 mm) column from Shodex (Japan) was used to separate the monosaccharides (Lareo et al., 2013). Glucose was detected by a low-temperature evaporative light-scattering detector (Sedex 80, ERB GmbH, Germany) as described by Clement, Yong, and Brechet (1992) with slight modifications. The mobile phase was 100% water, the flow rate 0.8 mL/min, column temperature 50 °C, detector gain 1, detector temperature 35 °C.

2.6. Polyphenol analysis

Aliquots of CPP (1 g), pre-treated materials (2.5 g) and CCR treated materials (2.5 g) were covered with aqueous formic acid (5 mL or 12.5 mL), rehydrated for 5 min (ice bath in the dark) and homogenized (Ultra Turrax, IKA-Werke, Germany). PP were extracted and anthocyanins, phenolic acids and flavonols were measured by HPLC-DAD (1100 series, Germany) at different wavelengths (520 nm, 325 nm and 360 nm, respectively) as described earlier (Mayer-Miebach et al., 2019). Total polyphenols contents (TPP) were determined using the Folin-Ciocalteu test and quantified using catechin monohydrate (Singleton, Orthofer, & Lamuela-Raventos, 1999).

PP contents of *in vitro* digested samples were centrifuged (17,000g, 20 °C, 10 min), filtered (0.22 μ m, Merck Millipore, Darmstadt, Germany), adjusted to 20% (v/v) formic acid and analyzed by HPLC according to a slightly modified method described previously (Marinelli et al., 2018). PP content was determined as sum of total anthocyanins, total flavonols and total phenolic acids. Bioaccessibility was calculated as the percentage of PP content measured in the samples after digestion relative to the PP content of the non-digested materials.

2.7. Analysis of antioxidant capacity

The AOX of CPP and CCR treated materials was assessed based on the ferric reducing antioxidant power (FRAP) assay according to Benzie and Strain (1996). The results are expressed as μ mol ferrous sulfate per g.

2.8. Residual moisture content

Residual moisture contents of all materials were determined by Karl-Fischer titration using an automatic titrator (Titroline alfa, Schott Instruments GmbH, Germany) (Bruttel & Schlink, 2006).

2.9. Statistics

Data of isolated DF (monosaccharide constituents and linkage, oligosaccharide screening) are reported as mean \pm range/2. Other data are presented as mean \pm standard deviation (SD). ANOVA followed by Holm-Sidak and Tukey-Kramer test was performed to determine statistical significance ($P < 0.05$) between groups using SigmaPlot software (version 13.0, Systat Software GmbH, Germany).

3. Results and discussion

3.1. Characterization of chokeberry pomace powder

The determined TDF content (57.8 ± 2.0 g/100 g dm) lies within the range of published data for chokeberry pomace and includes about 76% IDF, 20% HMW-SDF and 4% LMW-SDF (Table 2) (Borycka & Stachowiak, 2008; Nawirska & Uklańska, 2008; Reifner et al., 2019; Sójka et al., 2013). CPP contains a large amount of TPP (5.5 ± 0.2 g/100 g dm, as determined by the nonspecific Folin-Ciocalteu assay), with about 33% anthocyanins, 6% hydroxycinnamic acids and 3% flavonols (Table 3). The free glucose ($5.2 \pm 0.2\%$), fructose ($4.5 \pm 0.2\%$), sucrose ($0.4 \pm 0.2\%$), sorbitol (7.5 ± 0.4 g/100 g dm) and individual PP contents determined (Tables S2, S3-1, S3-2, S3-3, Supporting Information) are also within the range of published data (Kulling & Rawel, 2008; Reifner et al., 2019). Thus, the CPP used in this study is rich in DF and bioactive PP and also contains sorbitol, a natural low caloric sweetener.

3.2. Dietary fiber composition

So far, little is known about the DF composition of chokeberry pomace. Nawirska and Kwaśniewska (2005) basically assigned pectic polysaccharides, hemicelluloses and cellulose according to their solubility in different media after enzymatic starch degradation. Detailed structural studies include the determination of the monosaccharide composition after hydrolysis as well as the analysis of their linkage types and ratios in the polymer, thereby enabling the assignment of monosaccharides to chokeberry pomace DF polysaccharides. Furthermore, recent enzymatic profiling methods provide additional insights into pectic substructures. Thus, structural features being particularly susceptible to thermomechanical treatment may be identified based on this detailed structural study design. However, due to the fact that polysaccharide hydrolysis always results in an (unsteady) equilibrium

between liberation of constituent monosaccharides and the degradation of already liberated monomers it is not possible to accurately analyze all polysaccharide monomers as a whole. Thus, all results are given in molar ratios rather than absolute quantities. There are only a few close botanical relatives of chokeberry of which the dietary fiber composition has been described in detail in the past. However, both *Aronia melanocarpa* and *Malus domestica* (apple) are members of the Rosaceae subtribe Malinae bearing the same botanical fruit type (pome). Wefers, Floerchinger, and Bunzel (2018) described the mono- and oligosaccharide composition of non-starch polysaccharides (NSP) of 14 apple cultivars, applying all methods stated above. Thus, our data will be compared to data from apple in the following.

3.2.1. Dietary fiber monosaccharide composition

The monosaccharide composition of IDF polysaccharides (after sulphuric acid hydrolysis) is shown in Fig. 1. IDF consists of 42.3 mol% glucose, 26.4 mol% xylose as well as 3.5 mol% mannose and some (predominantly) pectic monosaccharides such as galacturonic acid, galactose and arabinose with 6.6 to 10.1 mol%. Due to the application of sulphuric acid hydrolysis, galacturonic acid portions are probably underestimated (methanolysis, however, would not capture cellulosic glucose). HMW-SDF mainly consists of galacturonic acid (24.6 mol%), arabinose (20.8 mol%) and galactose (14.7 mol%); hemicellulosic sugars such as xylose, mannose and glucose contribute with 39.8 mol% in total (after methanolysis). Thus, the IDF fraction appears to contain large amounts of cellulose but also significant amounts of xylose containing hemicelluloses such as xylans and xyloglucans and minor amounts of mannans. Approximately 26 mol% of the monosaccharides liberated from IDF after sulphuric acid hydrolysis were roughly assigned to pectic polymers such as homogalacturonan and type I rhamnogalacturonan with arabinose and galactose containing sidechains. HMW-SDF predominately consists of pectic polymers (64 mol% of the liberated monosaccharides after methanolysis were roughly assigned to pectic polysaccharides). Compared to NSP from apple fruits after sulphuric acid hydrolysis, CPP IDF shows a different monosaccharide composition. Most strikingly, xylose portions are much higher than in apple whereas galacturonic acid and arabinose portions are lower. However, it is important to keep in mind that NSP include HMW-SDF.

3.2.2. Dietary fiber polysaccharide linkage types

The profiles of partially methylated alditol acetates (PMAA) are given in Table 4. Accounting for 40.4 mol%, 1,4-linked glucopyranose units are dominant in IDF. Although a minor portion can be assigned to xyloglucans, the vast majority of 1,4-linked glucopyranose units represents cellulose. Arabinose (total of 25.8 mol%) is mostly represented by terminal and 1,5-linked arabinose units, the latter representing linear arabinan regions of neutral rhamnogalacturonan I side-chains, with fewer branched (1,3,5-/1,2,5-/1,2,3,5-linked) arabinofuranose units. Arabinans and galactans (represented by 1,4-galactopyranose

Table 2

Dietary fiber (DF) contents of chokeberry pomace powder (CPP), pre-treated material and samples after defined thermal and/or mechanical treatments (g/100 g dm, mean \pm SD, $n = 2$). T: temperature ($^{\circ}\text{C}$), $\dot{\gamma}$: shear rate (s^{-1}), t: time (min), c_w : water content (%), IDF: insoluble DF, HMW-SDF: high molecular weight soluble DF, LMW-SDF: low molecular weight soluble DF, TDF: total dietary fiber.

Sample	T	$\dot{\gamma}$	t	c_w	IDF ^a	HMW-SDF ^a	LMW-SDF ^a	TDF ^a
CPP	–	–	–	3.5 ± 0.6	43.8 ± 1.4^A	11.5 ± 1.2^{AB}	2.4 ± 2.2^A	57.8 ± 2.0^A
Pre-treated	–	–	–	12	50.2 ± 0.2^B	12.6 ± 0.7^{AB}	2.0 ± 0.2^A	64.8 ± 0.6^{AB}
B	100	50	1	12	49.6 ± 1.2^{AB}	10.1 ± 1.5^{AB}	0.9 ± 1.2^A	60.6 ± 3.9^A
C	140	0.1	1	12	46.1 ± 1.7^{AB}	12.1 ± 1.2^{AB}	2.6 ± 1.3^A	60.8 ± 0.9^A
D	140	12.5	1	12	47.0 ± 4.3^{AB}	14.7 ± 2.6^A	1.9 ± 0.0^A	63.6 ± 6.9^{AB}
E	140	50	1	12	48.9 ± 5.1^{AB}	10.0 ± 2.9^{AB}	1.0 ± 0.0^A	60.0 ± 2.2^A
F	140	50	1	22	45.6 ± 0.4^{AB}	15.1 ± 0.2^A	2.0 ± 0.3^A	62.7 ± 0.2^{AB}
G	140	50	20	12	51.8 ± 1.0^B	11.9 ± 0.2^{AB}	1.9 ± 0.0^A	65.6 ± 0.9^{AB}
H	160	50	1	12	41.9 ± 0.4^A	14.0 ± 0.3^A	0.8 ± 0.3^A	56.7 ± 0.1^A
I	160	50	20	12	61.7 ± 0.5^C	7.3 ± 0.8^B	2.5 ± 0.0^A	71.5 ± 0.3^B

^a Means with different superscript capital letters within the same column differ significantly ($P < 0.05$).

Table 3

Polyphenols content (PP) of chokeberry pomace powder (CPP), pre-treated material and samples after defined thermal and/or mechanical treatments (g/100 g dm, mean \pm SD, n = 2). T: temperature ($^{\circ}$ C), $\dot{\gamma}$: shear rate (s^{-1}), t: time (min), c_w : water content (%), TPP: total polyphenols.

Sample	T	$\dot{\gamma}$	t	c_w	Anthocyanins ^a	Phenolic acids ^a	Flavonols ^a	TPP ^a
CPP	–	–	–	3.5 \pm 0.6	1.80 \pm 0.09 ^A	0.31 \pm 0.03 ^A	0.184 \pm 0.006 ^A	5.5 \pm 0.2 ^A
Pre-treated	–	–	–	12	1.57 \pm 0.07 ^B	0.26 \pm 0.02 ^{AB}	0.196 \pm 0.022 ^A	5.2 \pm 0.2 ^{AB}
B	100	50	1	12	1.24 \pm 0.12 ^C	0.20 \pm 0.01 ^{BC}	0.111 \pm 0.006 ^B	3.6 \pm 0.1 ^{BC}
C	140	0.1	1	12	0.60 \pm 0.03 ^D	0.17 \pm 0.00 ^{CD}	0.115 \pm 0.002 ^{BC}	3.3 \pm 0.2 ^C
D	140	12.5	1	12	0.54 \pm 0.07 ^D	0.19 \pm 0.02 ^{BC}	0.142 \pm 0.017 ^{BD}	4.6 \pm 0.6 ^{AC}
E	140	50	1	12	0.63 \pm 0.10 ^D	0.18 \pm 0.02 ^C	0.090 \pm 0.007 ^{BE}	3.4 \pm 0.5 ^C
F	140	50	1	22	0.54 \pm 0.04 ^D	0.22 \pm 0.00 ^{BC}	0.159 \pm 0.001 ^{ACD}	5.8 \pm 0.1 ^A
G	140	50	20	12	0.02 \pm 0.00 ^E	0.09 \pm 0.03 ^{DEF}	0.054 \pm 0.014 ^{EPG}	1.2 \pm 0.3 ^D
H	160	50	1	12	0.24 \pm 0.03 ^F	0.16 \pm 0.04 ^{CE}	0.084 \pm 0.012 ^{BF}	3.8 \pm 1.3 ^{BC}
I	160	50	20	12	< LOQ	0.07 \pm 0.01 ^F	0.026 \pm 0.001 ^G	0.6 \pm 0.1 ^D

< LOQ: below limit of quantification.

^a Means with different superscript capital letters within the same column differ significantly (P < 0.05).

units) as neutral rhamnogalacturonan I side-chains are attached via 1,2,4-linked rhamnopyranose units to the backbone. Further relevant PMAA are 1,4-linked xylopyranose units, representing hemicellulosic xylans, 1,2-linked xylopyranose units from xyloglucan sidechains and terminal xylose units potentially originating from both xylans and xyloglucans (Scheller & Ulvskov, 2010). Additional monomers represent the xyloglucan backbone (1,4,6-linked glucopyranose units) and hemicellulosic mannans (1,4-linked mannopyranose units).

Due to the structure dependent water solubility of DF polysaccharides, the profile obtained from HMW-SDF largely differs from the IDF profile. Reflecting the absence of cellulose, arabinose units dominate in HMW-SDF (39.8 mol% of the PMAA), demonstrating the predominance of pectic polysaccharides in this DF fraction. Different from IDF, noticeable amounts of type II arabinogalactans (from arabinogalactan proteins) can be identified based on portions of 1,3,6-, 1,3- and 1,6-linked galactopyranose units. Just as described for IDF, xylose derivatives represent both xylans and xyloglucans.

Not surprisingly, the major polysaccharide linkage types were also reported for apple NSP (Wefers et al., 2018); however, some minor linkage types such as 1,3-Araf and 1,2-Araf were not quantitated in the mentioned study. On a more general note, interpretation of the methylation analysis data suggests that apple NSP appear to have higher portions of xyloglucans, but contain less xylan than both CPP IDF and CPP HMW-SDF. Also, differences in the arabinan branching patterns

were noted.

Methylation analysis in general confirms the assignment of mono-saccharides to the individual polysaccharides and provides additional details about structural features of these polysaccharides; however, the methylation analysis protocol as performed here allows for an analysis of neutral monomers only. Keeping this limitation in mind, methylation analysis shows that about a quarter of the neutral IDF monomers derive from pectic arabinan sidechains, with 1,5-, 1,3-, 1,3,5-, 1,2,5- and 1,2,3,5-linked arabinofuranose units that can be assigned to type I rhamnogalacturonan arabinans (Wefers & Bunzel, 2016). Comparing the relative amount of arabinan backbone containing structural units to the relative amount of 1,2,4-linked rhamnopyranose units demonstrates that IDF contains rather long arabinans as neutral type I rhamnogalacturonan sidechains. The same holds true for HMW-SDF pectins. Additional information on arabinan structural details was obtained from an enzymatic arabinan profiling.

3.2.3. Arabinan oligosaccharides

Arabino-oligosaccharides were liberated from IDF and HMW-SDF by an *endo*-arabinanase. This enzyme specifically cleaves linear 1,5-linked units within pectic arabinans, thereby releasing diagnostic oligosaccharides (Fig. 2). Ratios of these structural elements liberated from CPP DF are given in Table 5.

IDF arabinans are largely based on A-2a (81.2 mol%), A-5b (9.1 mol%

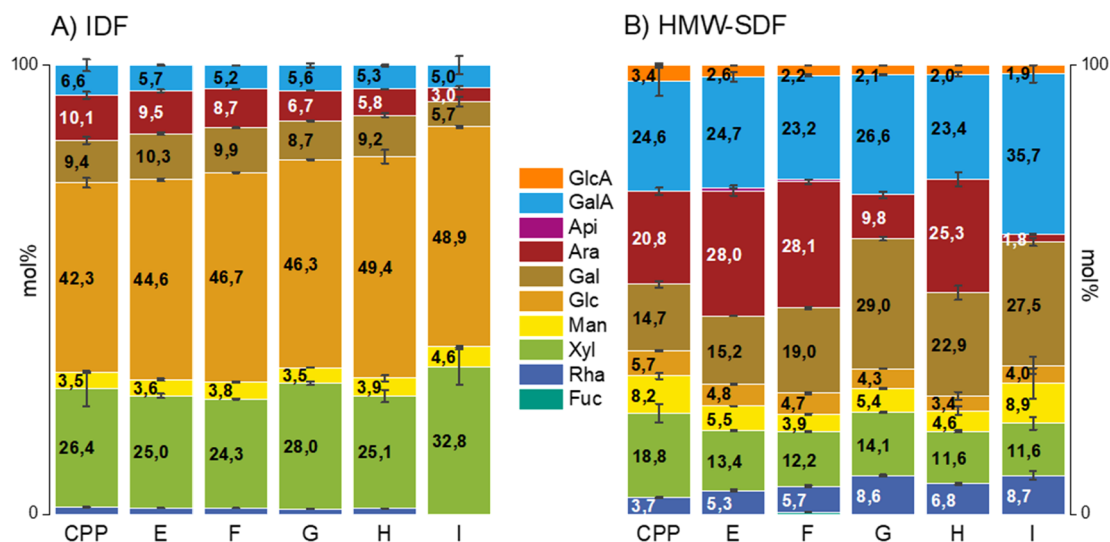


Fig. 1. Monosaccharide composition of (A) insoluble dietary fibre (IDF) and (B) soluble high molecular weight dietary fibre (HMW-SDF) from chokeberry pomace powder (CPP) before and after thermal treatment at constant shear rate ($50 s^{-1}$) (mol%, range/2, n = 2). E: 140 $^{\circ}$ C, 1 min, 12% water, F: 140 $^{\circ}$ C, 20 min, 12% water, G: 140 $^{\circ}$ C, 20 min, 12% water, H: 160 $^{\circ}$ C, 1 min, 12% water, I: 160 $^{\circ}$ C, 20 min, 12% water. GlcA: D-glucuronic acid, GalA: D-galacturonic acid, Api: D-apiiose, Ara: L-arabinose, Gal: D-galactose, Glc: D-glucose, Man: D-mannose, Rha: L-rhamnose, Xyl: D-xylose, Fuc: L-fucose.

Table 4
Partially methylated alditol acetates (PMAA) of insoluble dietary fiber (IDF) and high molecular weight soluble dietary fiber (HMW-SDF) fractions (mol%, mean \pm range/ $2, n = 2$) isolated from chokeberry pomace powder (CPP) before and after treatment at constant shear rate (50 s^{-1}) and different temperatures, times and water contents.^a

Glycosidic linkage ^b	IDF							HMW-SDF						
	CPP	E	F	G	H	I	CPP	E	F	G	H	I		
t-Araf	7.0 \pm 0.6	5.8 \pm 0.3	5.6 \pm 0.3	3.6 \pm 0.3	3.8 \pm 0.2	1.3 \pm 0.2	15.7 \pm 0.9	18.7 \pm 0.2	17.6 \pm 0.6	9.9 \pm 0.6	16.9 \pm 0.2	2.0 \pm 0.3		
t-Arap	0.9 \pm 0.1	0.9 \pm 0.1	0.9 \pm 0.1	1.0 \pm 0.1	1.2 \pm 0.1	0.6 \pm 0.1	1.9 \pm 0.1	1.1 \pm 0.1	1.6 \pm 0.1	1.9 \pm 0.1	1.2 \pm 0.1	1.6 \pm 0.3		
1,2-Araf	0.3 \pm 0.1	0.6 \pm 0.1	0.5 \pm 0.1	0.4 \pm 0.1	0.8 \pm 0.5	0.5 \pm 0.4	0.9 \pm 0.3	0.5 \pm 0.1	0.5 \pm 0.1	0.5 \pm 0.1	0.8 \pm 0.3	2.0 \pm 1.2		
1,3-Araf	1.8 \pm 0.3	2.2 \pm 0.2	1.7 \pm 0.1	1.4 \pm 0.1	2.1 \pm 0.7	1.2 \pm 0.5	3.3 \pm 0.4	4.6 \pm 0.3	4.5 \pm 0.2	3.3 \pm 0.2	4.5 \pm 0.4	5.4 \pm 2.0		
1,5-Araf	10.4 \pm 1.3	7.7 \pm 0.5	7.0 \pm 0.3	5.6 \pm 0.6	5.5 \pm 0.7	3.8 \pm 2.6	17.0 \pm 1.0	24.9 \pm 0.2	25.6 \pm 1.8	10.8 \pm 0.1	21.0 \pm 1.1	2.8 \pm 0.3		
1,2,5-Araf	0.8 \pm 0.4	0.3 \pm 0.1	0.3 \pm 0.1	0.3 \pm 0.1	0.2 \pm 0.1	0.1 \pm 0.1	0.7 \pm 0.1	1.0 \pm 0.1	0.9 \pm 0.1	0.5 \pm 0.1	1.0 \pm 0.1	ND		
1,3,5-Araf	3.9 \pm 0.2	2.6 \pm 0.2	2.6 \pm 0.1	1.4 \pm 0.1	1.7 \pm 0.3	0.5 \pm 0.4	3.1 \pm 0.2	7.1 \pm 0.2	7.0 \pm 0.5	2.1 \pm 0.1	7.0 \pm 0.1	0.5 \pm 0.1		
1,2,3,5-Araf	0.8 \pm 0.1	0.6 \pm 0.1	0.5 \pm 0.1	0.9 \pm 0.1	0.3 \pm 0.1	0.6 \pm 0.3	1.2 \pm 0.3	2.2 \pm 0.3	1.6 \pm 0.1	0.5 \pm 0.1	1.6 \pm 0.1	0.1 \pm 0.1		
Σ Ara	25.8 \pm 1.7	20.8 \pm 0.7	19.1 \pm 0.6	14.6 \pm 0.6	15.5 \pm 1.2	8.7 \pm 1.5	43.8 \pm 2.3	60.2 \pm 0.3	59.4 \pm 2.5	29.4 \pm 0.3	54.0 \pm 0.8	14.3 \pm 3.2		
t-Galp	3.1 \pm 0.3	4.1 \pm 0.2	3.7 \pm 0.2	4.2 \pm 0.4	5.1 \pm 1.3	5.8 \pm 2.6	4.7 \pm 0.5	4.1 \pm 0.2	4.6 \pm 0.1	7.1 \pm 0.1	5.2 \pm 0.1	10.5 \pm 0.4		
1,2-Galp	ND	ND	ND	ND	ND	ND	1.2 \pm 0.3	1.0 \pm 0.1	0.8 \pm 0.2	0.7 \pm 0.1	0.9 \pm 0.4	1.3 \pm 0.2		
1,3-Galp	0.2 \pm 0.1	0.2 \pm 0.1	0.2 \pm 0.1	0.2 \pm 0.1	0.3 \pm 0.1	0.2 \pm 0.1	1.8 \pm 0.1	1.5 \pm 0.1	1.2 \pm 0.3	1.7 \pm 0.1	1.4 \pm 0.1	1.2 \pm 0.4		
1,4-Galp	2.9 \pm 0.4	4.6 \pm 0.1	3.5 \pm 0.3	2.2 \pm 0.3	3.9 \pm 0.2	1.2 \pm 0.1	1.4 \pm 0.6	3.0 \pm 0.1	4.7 \pm 1.0	19.4 \pm 1.5	6.5 \pm 0.7	22.3 \pm 1.0		
1,6-Galp	0.7 \pm 0.1	0.9 \pm 0.1	0.8 \pm 0.1	1.2 \pm 0.1	1.0 \pm 0.1	0.9 \pm 0.1	2.2 \pm 0.2	1.3 \pm 0.1	1.3 \pm 0.1	3.4 \pm 0.2	1.4 \pm 0.2	4.7 \pm 0.8		
1,3,4-Galp	0.2 \pm 0.1	0.2 \pm 0.1	0.2 \pm 0.1	0.4 \pm 0.1	0.4 \pm 0.1	0.5 \pm 0.1	ND	ND	ND	ND	ND	ND		
1,3,6-Galp	0.7 \pm 0.1	0.7 \pm 0.1	0.6 \pm 0.1	0.4 \pm 0.1	0.5 \pm 0.1	0.1 \pm 0.1	6.5 \pm 0.8	4.7 \pm 0.2	4.1 \pm 0.9	4.0 \pm 0.2	3.2 \pm 1.0	1.5 \pm 0.5		
Σ Gal	7.8 \pm 0.7	10.7 \pm 0.1	8.9 \pm 0.5	8.6 \pm 0.8	11.2 \pm 1.2	8.7 \pm 3.0	17.8 \pm 1.0	15.6 \pm 0.6	16.7 \pm 2.4	36.2 \pm 1.0	18.6 \pm 1.5	41.6 \pm 2.9		
t-Glcp	1.1 \pm 0.3	1.1 \pm 0.1	1.2 \pm 0.2	1.8 \pm 0.1	1.2 \pm 0.2	1.2 \pm 0.1	1.7 \pm 0.6	1.2 \pm 0.1	0.9 \pm 0.1	0.8 \pm 0.1	1.1 \pm 0.2	1.3 \pm 0.2		
1,4-Glcp	40.4 \pm 0.1	43.3 \pm 0.1	44.9 \pm 0.7	44.5 \pm 0.5	45.8 \pm 4.0	40.0 \pm 2.2	8.8 \pm 1.0	3.9 \pm 0.1	4.7 \pm 0.1	5.0 \pm 0.1	5.8 \pm 2.2	8.9 \pm 2.6		
1,4,6-Glcp	3.7 \pm 0.4	3.8 \pm 0.4	3.9 \pm 0.1	4.4 \pm 0.4	4.2 \pm 1.0	3.8 \pm 0.6	2.8 \pm 0.6	1.6 \pm 0.1	1.9 \pm 0.2	3.8 \pm 0.1	2.0 \pm 0.1	3.5 \pm 1.1		
Σ Glc	45.2 \pm 0.2	48.2 \pm 0.4	50.0 \pm 0.4	50.7 \pm 0.8	51.3 \pm 4.8	45.0 \pm 1.8	13.3 \pm 2.1	6.7 \pm 0.1	7.5 \pm 0.3	9.6 \pm 0.1	8.9 \pm 2.3	13.6 \pm 1.7		
t-Manp	0.2 \pm 0.1	0.2 \pm 0.1	0.2 \pm 0.1	0.2 \pm 0.1	0.2 \pm 0.1	0.2 \pm 0.1	2.4 \pm 1.3	1.4 \pm 0.1	0.7 \pm 0.1	1.0 \pm 0.2	1.3 \pm 0.4	2.1 \pm 0.9		
1,4-Manp	2.6 \pm 0.4	3.2 \pm 0.1	3.2 \pm 0.3	3.4 \pm 0.2	3.5 \pm 0.6	3.4 \pm 0.3	2.2 \pm 0.4	1.0 \pm 0.2	1.4 \pm 0.1	1.5 \pm 0.1	1.1 \pm 0.2	1.8 \pm 0.4		
1,4,6-Manp	0.5 \pm 0.1	0.6 \pm 0.1	0.6 \pm 0.1	0.7 \pm 0.1	0.6 \pm 0.2	0.6 \pm 0.2	0.8 \pm 0.2	0.8 \pm 0.2	ND	ND	ND	ND		
Σ Man	3.3 \pm 0.4	4.0 \pm 0.1	4.0 \pm 0.3	4.4 \pm 0.3	4.3 \pm 0.8	4.1 \pm 0.6	5.4 \pm 1.8	2.4 \pm 0.1	2.1 \pm 0.1	2.5 \pm 0.3	2.4 \pm 0.5	3.9 \pm 0.5		
1,2-Rhap	0.6 \pm 0.3	0.7 \pm 0.1	0.7 \pm 0.1	0.8 \pm 0.1	0.4 \pm 0.1	0.4 \pm 0.1	0.8 \pm 0.1	1.6 \pm 0.1	1.4 \pm 0.1	2.4 \pm 0.2	1.9 \pm 0.1	3.5 \pm 0.4		
1,2,4-Rhap	0.3 \pm 0.1	0.5 \pm 0.1	0.5 \pm 0.1	0.7 \pm 0.2	0.4 \pm 0.1	0.2 \pm 0.1	0.4 \pm 0.1	0.6 \pm 0.1	0.8 \pm 0.1	1.2 \pm 0.1	0.9 \pm 0.1	1.5 \pm 0.4		
Σ Rha	0.9 \pm 0.4	1.2 \pm 0.1	1.2 \pm 0.1	1.5 \pm 0.2	0.8 \pm 0.1	0.5 \pm 0.1	1.2 \pm 0.1	2.2 \pm 0.2	2.2 \pm 0.2	3.6 \pm 0.2	2.8 \pm 0.1	5.0 \pm 0.8		
t-Xylp	5.0 \pm 0.1	6.3 \pm 0.2	5.6 \pm 0.1	6.7 \pm 1.3	6.2 \pm 0.8	5.3 \pm 0.7	4.3 \pm 0.5	3.9 \pm 0.2	4.3 \pm 0.3	6.4 \pm 0.1	5.1 \pm 0.1	8.8 \pm 0.3		
1,2-Xylp ^c	2.1 \pm 0.1	2.1 \pm 0.1	2.4 \pm 0.1	2.0 \pm 0.3	2.3 \pm 0.1	3.0 \pm 0.4	1.8 \pm 0.3	1.5 \pm 0.2	1.4 \pm 0.1	2.8 \pm 0.1	1.6 \pm 0.2	2.7 \pm 0.5		
1,4-Xylp ^c	9.8 \pm 3.6	6.8 \pm 1.0	8.7 \pm 0.5	11.4 \pm 2.0	8.5 \pm 2.4	24.6 \pm 3.0	12.3 \pm 0.7	7.4 \pm 0.5	6.3 \pm 0.4	9.4 \pm 0.3	6.6 \pm 0.3	10.1 \pm 1.4		
Σ Xyl	17.0 \pm 3.5	15.2 \pm 1.2	16.8 \pm 0.6	20.2 \pm 0.4	17.0 \pm 3.2	33.0 \pm 2.8	18.5 \pm 0.5	12.8 \pm 0.9	12.1 \pm 0.1	18.6 \pm 0.1	13.3 \pm 0.4	21.6 \pm 1.7		

ND: not detected.

^a E: 14 °C, 1 min, 12% water, F: 140 °C, 1 min, 22% water, G: 140 °C, 20 min, 12% water, H: 160 °C, 1 min, 12% water, I: 160 °C, 20 min, 12% water.

^b Ara: arabinose, Gal: galactose, Glc: glucose, Man: mannose, Rha: rhamnose, Xyl: xylose, f: furanose, p: pyranose.

^c Coeluting, calculated from the area ratio of characteristic fragment ion peaks. *m/z* 117: 1,2-Xylp, *m/z* 118: 1,4-Xylp.

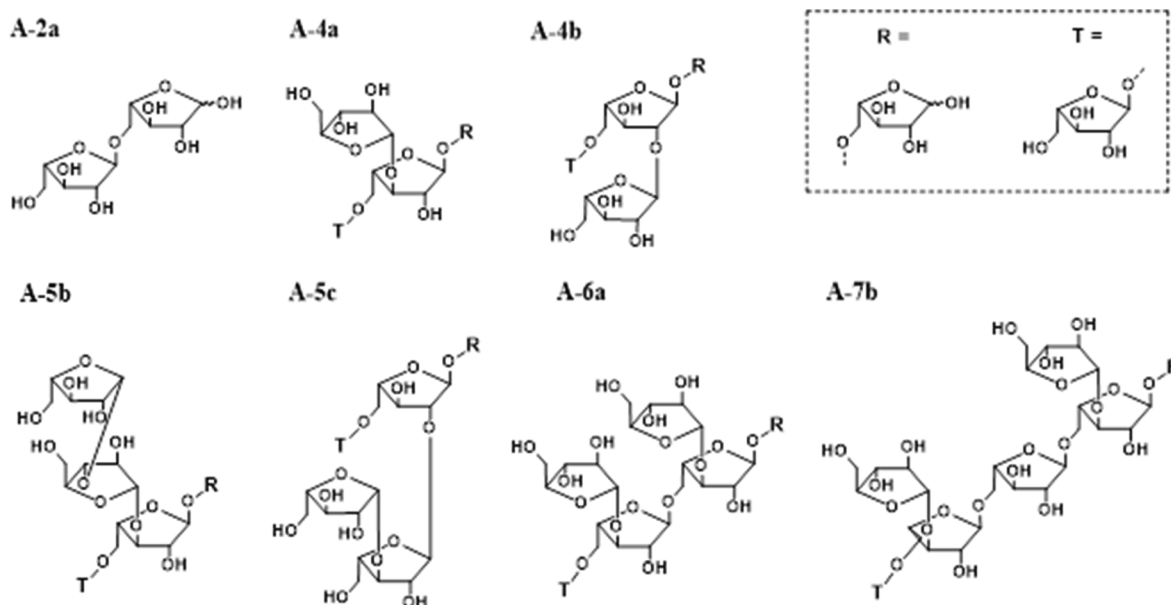


Fig. 2. Identified pectic arabinan oligosaccharides of dietary fiber fractions isolated from chokeberry pomace powder and thermomechanically treated materials after enzymatic cleavage by *endo*-arabinanase. T: terminal end, R: reducing end.

% and A-4a (5.1 mol%) oligosaccharides. A-5b represents a less common structural feature of arabinans. This structural element (just as A-5c) also contributes to the 1,3-linked arabinofuranose units as determined by methylation analysis. Also, the detection of A-4a, A-6a and A-7b agrees with the methylation analysis data demonstrating the predominance of (single residue) side-chains attached in O3-position over side-chains attached in O2-position (represented by A-4b) to the backbone. Both, A-7b but more importantly A-6a demonstrate clusters of branches within these generally moderately branched arabinans. The arabinan motives of CPP HMW-SDF appear to be identical with IDF arabinans; however, oligosaccharide A-5c cannot be quantified (Table 5). This has also been observed for apple NSP where A-5c was not quantifiable before storage (Wefers et al., 2018).

3.3. Effect of thermomechanical treatment on dietary fiber structure and content

Comparing the monosaccharide composition of DF polysaccharides in CPP with the material treated either thermally and/or mechanically provides valuable hints at fragile structures in DF polysaccharides.

3.3.1. Monosaccharide constituents

IDF monosaccharide compositions of CCR treated CPP with a water content of 12% is largely comparable to the untreated material after short-time and moderate temperature treatment (E: 140 °C, 1 min). After 20 min at 160 °C (I), more pronounced alterations are detected: galactose, galacturonic acid and arabinose portions decrease (from 26.1 to 13.7 mol% in total), portions of glucose and xylose increase (Fig. 1A). The monosaccharide composition of HMW-SDF shows more distinct changes in response to the thermomechanical treatment (Fig. 1B). After 20 min at 160 °C (I), arabinose and xylose portions strongly decrease from 20.8 mol% to 1.8 mol% and from 18.8 mol% to 11.6 mol%, respectively. Water content, 12% or 22%, does not affect monosaccharide compositions in either IDF or HMW-SDF during treatment at 140 °C for 1 min (E, F).

3.3.2. Dietary fiber polysaccharide linkage type

Data from linkage analysis of IDF and HMW-SDF polysaccharides after thermomechanical treatment are given in Table 4. Considering the IDF fraction of treated samples, only minor to moderate modifications

result from short-time treatment at 140 °C (E). However, it is obvious that especially portions of terminal, linear (1,5-/1,3-linked) and branched (1,2,3-/1,3,5-/1,2,3,5-linked) arabinofuranose units start to decline. This degradation continues with increased thermomechanical stress until, after a treatment for 20 min at 160 °C (I), merely 7.5 mol% of the initial 24.7 mol% is left. The portion of 1,4-linked glucopyranose units remains largely constant during treatment, whereas the portion of 1,4-linked xylopyranose units increases from 9.8 mol% to 24.6 mol% at 160 °C and prolonged residence time (I). Thermomechanical treatment appears to have a greater impact on HMW-SDF from CPP. After 1 min at 140 °C (E), the sum of arabinofuranose monomers increase. However, regarding long-time treatment at 160 °C (I), the arabinose components largely decrease with the exception of terminal arabinopyranose units and 1,3-linked arabinofuranose units. At the same time, both the portion of 1,4-linked galactopyranose and terminal galactopyranose units rise from 1.4 mol% to 22.3 mol% and from 4.7 mol% to 10.5 mol%, respectively.

3.3.3. Arabinan oligosaccharides

Ratios of the structural elements liberated from DF of treated samples are given in Table 5. Considering thermomechanically treated IDF samples, no distinct changes are caused by short-time treatments (E: 140 °C, 1 min). With increased temperature and/or time, minor oligosaccharides are no longer quantifiable. After 20 min at 160 °C (I), A-2a can be quantified in IDF hydrolysates. In contrast to IDF arabinans, most of the arabinan motives initially liberated from HMW-SDF arabinans are not even detectable after the treatment for 20 min at 160 °C (I); this also holds true for the originally predominant motive A-2a.

Our data indicate that especially arabinose containing polymers in IDF and HMW-SDF represent labile entities in terms of thermomechanical stress as indicated by the portions of arabinose units in both monosaccharide composition data and methylation analysis data. In case of IDF samples, portions of this monosaccharide strongly decrease, already starting at short-time treatments. Modifications of characteristic type I rhamnogalacturonan sidechains, arabinans, are assumed as supported by methylation analysis data and data obtained from the enzymatic arabinan profiling. In HMW-SDF, short-time treatments lead to higher arabinose portions initially, whereas long-time treatments cause dramatic reductions. This appears not logical at first glance; however, as the thermomechanical treatment may also alter DF

Table 5
Relative composition of pectic arabinan oligosaccharides liberated from chokeberry pomace powder (CPP) and treated dietary fiber (DF) fractions^a after enzymatic cleavage by *endo*-arabinanase (mol%, mean \pm range/ $n = 2$). IDF: insoluble dietary fiber, HMW-SDF: high molecular weight soluble dietary fiber.

Compound	IDF							HMW-SDF						
	CPP	E	F	G	H	I	CPP	E	F	G	H	I		
A-2a	81.2 \pm 0.1	77.8 \pm 0.4	77.5 \pm 0.2	84.8 \pm 0.1	79.7 \pm 0.2	100.0 \pm 0.1	84.9 \pm 0.9	85 \pm 0.4	85.1 \pm 0.2	89.2 \pm 0.4	84.7 \pm 0.1	< LOQ		
A-4a	5.1 \pm 0.1	7.4 \pm 0.2	8.0 \pm 0.2	10.9 \pm 0.1	8.6 \pm 0.5	< LOQ	5.4 \pm 0.4	5.4 \pm 0.2	5.3 \pm 0.1	10.8 \pm 0.4	6.1 \pm 0.1	< LOQ		
A-4b	0.9 \pm 0.1	1.0 \pm 0.1	1.0 \pm 0.1	< LOQ	1.1 \pm 0.1	< LOQ	1.1 \pm 0.1	1.3 \pm 0.1	1.2 \pm 0.1	< LOQ	1.2 \pm 0.1	< LOQ		
A-5b	9.1 \pm 0.1	8.9 \pm 0.1	8.5 \pm 0.1	4.3 \pm 0.1	7.6 \pm 0.6	< LOQ	7.6 \pm 0.1	7.1 \pm 0.2	7.1 \pm 0.1	< LOQ	6.4 \pm 0.1	< LOQ		
A-5c	2.4 \pm 0.1	2.4 \pm 0.1	2.2 \pm 0.1	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ		
A-6a	0.6 \pm 0.1	1.1 \pm 0.1	1.3 \pm 0.1	< LOQ	1.3 \pm 0.1	< LOQ	0.4 \pm 0.4	0.6 \pm 0.1	0.6 \pm 0.1	< LOQ	0.8 \pm 0.1	< LOQ		
A-7b	0.7 \pm 0.1	1.4 \pm 0.1	1.5 \pm 0.1	< LOQ	1.7 \pm 0.2	< LOQ	0.6 \pm 0.1	0.6 \pm 0.1	0.6 \pm 0.1	< LOQ	0.9 \pm 0.1	< LOQ		

< LOQ: below limit of detection, < LOQ: below limit of quantification.

^a E: 140 °C, 1 min, 12% water, F: 140 °C, 1 min, 22% water, G: 140 °C, 20 min, 12% water, H: 160 °C, 1 min, 12% water, I: 160 °C, 20 min, 12% water.

solubility in water and/or ethanol it also has to be considered that specific polymers may not be (fully) degraded but captured in a different DF fraction.

In contrast to arabinose containing structural units, 1,4-linked xylopyranose portions of IDF and terminal as well as 1,4-galactopyranose portions of HMW-SDF tend to increase with increasing thermomechanical stress, suggesting that galactans and xylans are more stable against thermomechanical treatment than arabinans.

Thus, we here report the first detailed analysis of CPP DF. In addition, we were able to demonstrate that individual polysaccharide substructures differ in their susceptibility towards degradation by thermomechanical stress.

3.3.4. Individual and total dietary fiber contents

Limited stability of DF polysaccharides under thermomechanical stress may also be reflected in the DF contents. However, both the individual DF contents (IDF, HMW-SDF and LMW-SDF) as well as the TDF contents do not follow a general trend (Table 2). No changes of the TDF or individual DF contents were detected after thermal stress at 100 °C or 140 °C (B, E) or different levels of mechanical stress at 140 °C (C–E) thus reflecting that no distinct changes in IDF and HMW-SDF structures were observed during thermomechanical treatments under these conditions. The highest total DF content was analyzed for the sample that experienced maximum thermomechanical stress (I: 160 °C, shear rate of 50 s⁻¹, 20 min). Although the content of HMW-SDF was reduced the IDF content was largely increased, resulting in the highest total DF content among the samples. Whereas the decreased HMW-SDF content reflects the limited stability of specific DF polysaccharides (see above) the increased IDF content is not verified by structural changes and may rather be explained by the thermally induced formation of other insoluble polymers, e.g. Maillard products such as melanoidins, that contribute to the enzymatically-gravimetrically analyzed IDF content. The formation of Maillard products from DF contributing to an increased IDF content in bread as compared to the wheat flour has been also reported (Pérez-Jiménez, Díaz-Rubio, Mesías, Morales, & Saura-Calixto, 2014). Considering the High-Temperature Short-Time extrusion processing, where the material is subjected to high temperatures (e.g. 140 °C) typically less than 30 s, no remarkable alterations in DF fractions and contents are to be expected. However, DF contents and structural changes need to be verified during High-Temperature Short-Time extrusion processing.

3.4. Sugar contents and glucose release after thermomechanical treatment and *in vitro* digestion

3.4.1. AOAC digests

Thermal treatment at 100 °C, 140 °C and 160 °C (B, E, H) leads to a reduction of glucose (5%, 20%, 30%) and fructose (5%, 15%, 20%). After 20 min at 160 °C (I) only 10% of the initial glucose and 5% of the initial fructose were detected. Sorbitol contents remain unchanged (Table S2, Supporting information).

3.4.2. *In vitro* gastrointestinal model

Bioaccessibility data of glucose from CPP after *in vitro* digestion is given in Fig. 3. Thermomechanical treatment at 80 °C for 1 min (A) does not significantly affect glucose bioaccessibility as compared to CPP, while treatment at 140 °C (E) reduces the bioaccessibility of glucose only slightly, at 160 °C for 1 min (H) by about 49% and at 160 °C for 20 min (I) nearly completely. These results are in line with the glucose losses as detected in the AOAC digests. Treatments at 80 °C show no effect on glucose release, but increasing temperature reduces the glucose content and consequently the amount of bioaccessible glucose released during *in vitro* digestion. Reduced glucose and fructose contents resulting from thermomechanical treatment at temperatures at about and above 140 °C indicate the formation of Maillard reaction products known to account for sensory properties and nutritional value

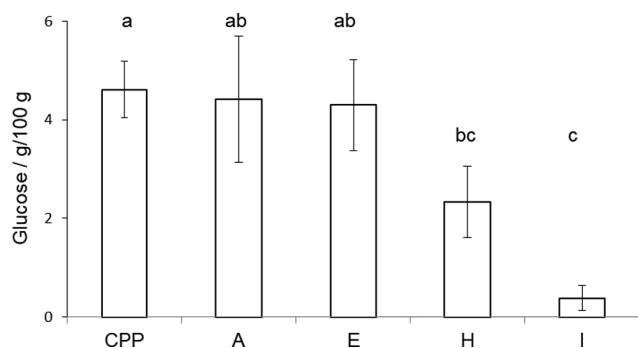


Fig. 3. Effect of thermal treatment of chokeberry pomace powder (CPP) at different temperatures and times on glucose release; glucose contents (mean \pm SD, $n \geq 3$). CPP: chokeberry pomace powder, A: 80 °C, 1 min, E: 140 °C, 1 min, H: 160 °C, 1 min, I: 160 °C, 20 min. Data with different letters are significantly different ($P < 0.05$).

of extruded products (Cheftel, 1986; Saldanha do Carmo et al., 2019). In particular, melanoidins e.g., generated during the last stage of the Maillard reaction and possibly exhibiting beneficial effects such as e.g. antioxidant capacity, are described as functional food ingredients (Mesías & Delgado-Andrade, 2017; Wang, Qian, & Yao, 2011). In view of possible beneficial health effects, the resulting reduced glucose contents together with the potential formation of Maillard products makes the CPP a suitable and promising material for extrusion processing.

3.5. Polyphenols stability and bioaccessibility

3.5.1. Stability after thermomechanical treatment

As compared to CPP, TPP and flavonol contents remain unchanged in pre-treated materials, whereas anthocyanins and phenolic acids are slightly reduced by about 10% (Table 3), presumably due to oxidative degradation and nucleophilic attack of water (Jackman, Yada, Tung, & Speers, 1987; Rodriguez-Amaya, 2019). However, hydration pre-treatment that is essential for CCR treatment is not used in extrusion processing. Thus, pre-treatment-based degradation of polyphenols does not appear to be relevant for extrusion processing.

Thermal treatment for 1 min additionally reduces the TPP content (about 40%), largely independent of variations in temperature and shear rate (B–E, H). After long-time treatments (20 min) TPP contents strongly decrease by 80% and 90% at 140 °C and 160 °C (G, I), respectively. High water content in the pre-treated material (22%) (F) leads to an improved PP retention by about 10%.

After thermal treatment at 100 °C for 1 min (B) total contents of anthocyanins, phenolic acids and flavonols are reduced by about 20%, 20% and 40%, respectively, as compared to the pre-treated material. At

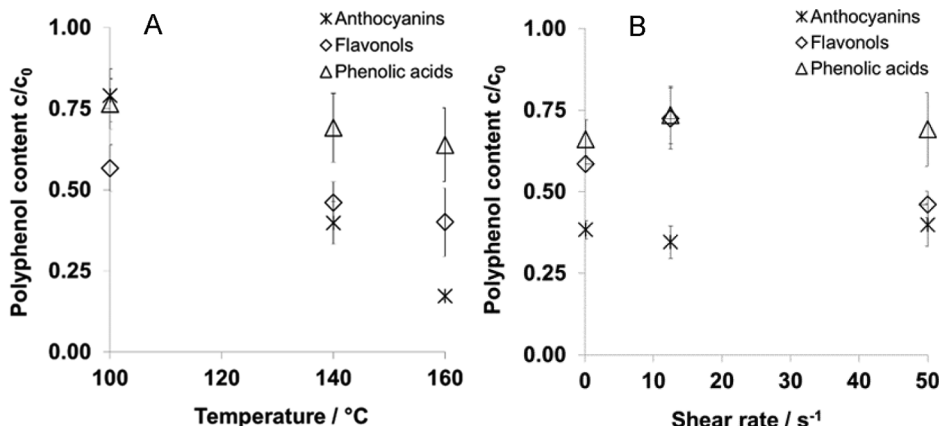


Fig. 4. Thermal (A) and mechanical (B) impact on polyphenol stability of treated materials (c_0 : based on chokeberry pomace powder (CPP) after pre-treatment, mean \pm SD, $n = 2$); A: constant shear rate 50 s^{-1} , treatment time 1 min, water content 12%, B: constant temperature 140 °C, treatment time 1 min, water content 12%

elevated temperatures (E, H: 140 °C, 160 °C) anthocyanins but not phenolic acids and flavonols are strongly degraded (60%, 85%). About 40% of the phenolic acids and 55% of the flavonols are retained at 160 °C (H) (Fig. 4A). Application of both temperatures combined with long-time treatments for 20 min (G, I) results in complete anthocyanin degradation and strongly reduced phenolic acid (65% and 75% reduction, respectively) and flavonol (70% and 90% reduction, respectively) contents (Table 3). Strong degradation of thermolabile anthocyanins and to a much lesser extent of phenolic acids and flavonols during extrusion processing of fruit materials mixed with starch is mainly due to combined thermal and mechanical effects (Camire et al., 2007; Hirth et al., 2015; Leonard et al., 2020; Leyva-Corrala et al., 2016; White et al., 2010). Moisture and starch content may prevent e.g. anthocyanins from degradation. As expected, the CPP contents of TPP, anthocyanins, phenolic acids and flavonols decrease with increasing thermal stress applied. Increasing mechanical stress, however, has no appreciable impact on the phenolic components (Fig. 4B). Effects of thermal and/or mechanical treatment on phenolic acid derivatives and on glycosides of anthocyanins and flavonols are given in Tables S3-1, S3-2, S3-3 (Supporting Information).

3.5.2. Bioaccessibility

PP (sum of anthocyanins, flavonols and hydroxycinnamic acids) of the untreated CPP are fully bioaccessible indicating their release from the chokeberry fiber matrix after consecutive gastric and intestinal *in vitro* digestion as described for maqui berries (Viuda-Martos et al., 2018). Bioaccessibility remains unchanged after short-time treatment up to 160 °C (A, E, H). However, long-time treatment at 160 °C (I) seems to reduce bioaccessibility by about 14% (Table 6, I). Though thermomechanical treatment reduces the PP content (Table 3), it hardly affects PP bioaccessibility, suggesting that digestion rather than thermomechanical treatment induces the release as already described for apple pomace (Liu et al., 2019). In order to combine, the fraction of bioaccessible PP after *in vitro* digestion were also calculated based on the initial PP content of CPP (Table 6, II). Data show that about 76% of the PP contained in CPP are bioaccessible after treatment at conditions relevant for extrusion processing (E, short-time at 140 °C). Here, about 35% of anthocyanins, 58% of phenolic acids and 49% of flavonols are retained (Table 3) and completely bioaccessible. Therefore, CPP can be considered for the fortification of extruded products with PP if appropriate temperature and treatment time are realized during extrusion processing.

3.6. Antioxidant activity

Table 6 shows the AOX of CPP and thermomechanically treated materials before and after *in vitro* digestion. Thermomechanical treatment up to 140 °C for 1 min (E) does not affect AOX as compared to the CPP. In contrast, treatment at 160 °C (H, I) decreases AOX by about 3

Table 6

Relative bioaccessibility of polyphenols (PP) after *in vitro* digestion (reference PP content: I – after *in vitro* digestion; II – CPP) and antioxidant activity (AOX) before and after *in vitro* digestion of chokeberry pomace powder (CPP) and samples after thermomechanical treatment.^a

Sample	T/°C	t/min	Bioaccessibility ^b		AOX ^c	
			I ^d	II ^d	Before <i>in vitro</i> digestion ^d	After <i>in vitro</i> digestion ^d
CPP	–	–	100 ± 5 ^A	100 ± 5 ^A	1111 ± 241 ^A	1110 ± 224 ^A
A	80	1	97 ± 4 ^A	100 ± 4 ^A	1031 ± 30 ^A	1089 ± 50 ^A
E	140	1	95 ± 1 ^A	76 ± 1 ^B	1296 ± 79 ^A	1172 ± 108 ^A
H	160	1	100 ± 4 ^A	19 ± 1 ^C	383 ± 49 ^A	523 ± 21 ^B
I	160	20	86 ± 15 ^A	2 ± 0.4 ^D	363 ± 45 ^B	294 ± 11 ^B

^a A, E, H, I: shear rate 50 s⁻¹, water content 12%.

^b Sum of anthocyanins, flavonols, hydroxycinnamic acids; expressed as %, mean ± SD, n ≥ 3.

^c Expressed as μmol Fe²⁺/g; mean ± SD, n ≥ 3.

^d Means with different superscript capital letters within the same column differ significantly (P < 0.05).

times irrespective of the treatment time. A similar trend is observed for the bioaccessible fractions after *in vitro* digestion. Furthermore, there is no difference in AOX of materials before and after *in vitro* digestion indicating that most of the antioxidants are released from the treated matrix during *in vitro* digestion and accumulate within the bioaccessible fraction. Although reduced AOX may be expected due to the degradation of PP during treatment at 140 °C (E), it is not significantly affected. Otherwise, increases in AOX as an effect of thermal processing are described (White et al., 2010). This is a further indication of Maillard products, which together with the retained PP may play a major role in AOX of CPP after thermomechanical treatment (Leonard et al., 2020; Mesias & Delgado-Andrade, 2017; White et al., 2010; Yilmaz & Toledo, 2005). The reduced glucose contents due to thermomechanical treatment and enhanced IDF after thermomechanical long-time treatment at 160 °C (I) also support this assumption. Yet, it does not explain the AOX reduction observed here.

4. Conclusion

Data from this study suggest that CCP is a good source of DF with the potential to partially substitute starch in extruded cereal products, e.g. breakfast cereals, thereby lowering the glycaemic response *in vitro*. Detailed study indicates that DF structure is basically not susceptible to thermomechanical stresses if applied in the typical range used for High-Temperature Short-Time extrusion processing. Under these conditions, an enrichment with completely bioaccessible PP is achieved simultaneously, although the content of PP is reduced with increasing thermal treatment; mechanical treatment has no impact on PP stability. Furthermore, AOX is not significantly affected by PP degradation. Long-time treatment, however, providing high thermomechanical stress, increases IDF content, reduces glucose content and release, and enhances AOX despite polyphenol degradation most likely all due to the formation of Maillard products, which are nowadays considered as potential functional food ingredients. Since optimum real processing conditions cannot be derived just from the results here presented, studies towards the impact of High-Temperature Short-Time extrusion processing on the DF and bioactive PP of CPP and mixtures with starch, thereby applying a pilot-scale twin-screw extruder and taking techno-functional properties into account, are currently ongoing.

CRedit authorship contribution statement

Vera Schmid: Conceptualization, Methodology, Supervision, Writing - review & editing, Investigation, Visualization, Writing - original draft. **Jan Steck:** Investigation, Visualization, Writing - original draft. **Esther Mayer-Miebach:** Project administration, Conceptualization, Methodology, Supervision, Writing - review & editing, Investigation, Visualization, Writing - original draft. **Diana Behnlian:** Conceptualization, Methodology, Supervision, Writing -

review & editing, Investigation, Visualization, Writing - original draft. **Karlis Briviba:** Conceptualization, Methodology, Supervision, Writing - review & editing, Investigation, Visualization, Writing - original draft. **Mirko Bunzel:** Conceptualization, Methodology, Supervision, Writing - review & editing. **Heike P. Karbstein:** Conceptualization, Methodology, Supervision, Writing - review & editing. **M. Azad Emin:** Project administration, Conceptualization, Methodology, Supervision, Writing - review & editing.

Declaration of Competing Interest

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

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Appendix A. Supplementary material

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