**ORIGINAL PAPER** 



# Thermo-mechanical processing of fibre-rich blackcurrant pomace to modify techno-functional properties

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Received: 22 March 2022 / Revised: 5 May 2022 / Accepted: 8 May 2022 / Published online: 4 June 2022 © The Author(s) 2022

## Abstract

Exploring the use of seedless blackcurrant pomace, a fibre-rich by-product of juice pressing, in foods is favourable due to its nutritional profile but also for economic and sustainability aspects. Current applications are limited to products in which rapid fibre swelling, high water solubility or low sedimentation is not essential. In this study, functional properties of seedless blackcurrant pomace were modified by thermo-mechanical treatments using extrusion cooking or micronization in a planetary ball mill. A full factorial design showed that low pomace moisture (11 g/100 g) had the highest impact on swelling capacity (+20.6%) and water solubility index (+23.2%), whereas variation in extrusion temperature exhibited only minor effects. After milling for 4 h, the median particle size was reduced by 98% to 4  $\mu$ m and the specific surface area increased from 0.1 to 2.5 m<sup>2</sup>/mL. Swelling capacity was highest after this time with 7.6 mL/g pomace and, although the amount of extractable sugars was reduced, water solubility increased to 7.6 g/100 g. In contrast to extruded samples, the red colour of the pomace was intensified after milling. Both treatments appear as promising to extend the applicability of fruit by-products in foods, as micronized pomace may counteract sedimentation in liquids, whereas increased swelling capacity after extrusion may have stabilizing effects on yoghurt-like systems.

Keywords By-product · Extrusion · Planetary ball milling · Techno-functional · Dietary fiber · Micronization

# Introduction

To meet the demand for food of a growing world population, an efficient and comprehensive utilisation of agricultural products is necessary. Currently, large amounts of by-products from fruit and vegetable processing are still discarded [1]. In the case of berry juice processing, approximately 25% of the raw material remains as pomace. This highly perishable material, which mainly consists of dietary fibre, can be used comprehensively after drying and milling, for instance in cereal-based products [2–5]. The applicability of such a material may be expanded when more valuable compounds are separated and exploited for their nutritional and techno-functional properties. A first low-effort and, at the same time, low-waste generating fractionation step is the mechanical separation of the seeds, which can be further

Anne-Marie Reißner anne-marie.reissner@tu-dresden.de used for oil extraction. Consequently, the dietary fibre content in the remaining pomace increases [6, 7].

Desired techno-functional properties, such as rapid swelling, high water-binding capacity, or sufficient water solubility, are basically determined by composition, structure, conformation, and physico-chemical characteristics of a material [8]. Different treatments may be applied to modify and optimise these properties. In contrast to chemical treatments, such as carboxymethylation or enzymatic-assisted techniques, thermo-mechanical processing is considered to be more environmentally friendly [9]. For fibre-rich materials, high-temperature short-time (HTST) extrusion can be used to change fibre structure and to increase water solubility. High temperature and shear forces in combination with high-pressure gradients on material exiting the extruder support the breakage of glycosidic bonds and result in the degradation of insoluble dietary fibre (IDF). These mechanisms were successfully applied to enhance techno-functional properties of pectin-rich fruit residues (citrus peels, apple pomace) [10–12], residues of protein providing kernels (soybeans, lupin fibre) [13–15] or bran [16, 17]. Optimum extrusion conditions in terms of high impact on the dietary

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fibre depended strongly on the respective material and the interactions between feed moisture, temperature, and screw speed. Therefore, they are difficult to predict in advance.

Fibre-rich residues can also be modified by superfine grinding (per definition to below 100  $\mu$ m in size [18]). Compared to knife mills or impact mills, the combination of centrifugal and Coriolis forces in planetary ball mills or the jet stream in jet mills leads to higher effectivity in particle size reduction [9]. Techno-functional properties change with size and surface characteristics of particles, but a high mechanical stress in such a mill also triggers chemical reactions. For instance, it was shown that the content of soluble dietary fibre (SDF) increased after micronization of okara and olive pomace, whereas IDF decreased in the same order of magnitude [19, 20]. Fourier-transform infrared spectroscopy indicated the breakage of intramolecular hydrogen bonds in cellulose and hemicellulose, which were exposed to mechanical degradation after degradation of the lignocellulosic fibre complex [20]. The cleavage of some  $\beta$ -1.4 glycosidic linkages of cellulose resulted in the formation of oligosaccharides [19]. Recent studies, where fibre was treated mechanically, focused mostly on vegetable or cereal by-products [18–21].

To the best of our knowledge, extrusion experiments on berry pomace were always conducted with additives (starch or other cereal components, e.g. [22, 23]) and, except for apple pomace [24], little is known on the techno-functional properties of micronized currant pomace. The aim of this study was to achieve knowledge whether techno-functional properties of seedless blackcurrant pomace (SBC) can be modified without supplements by such a thermo-mechanical treatment, and which extrusion parameters contribute to modification. Planetary ball milling was chosen as a second treatment.

# **Materials and methods**

## **Pomace preparation**

Blackcurrant pomace was provided by Austria Juice GmbH (Allhartsberg, Austria). Before juicing in hydraulic belt presses, the berries were enzymatically treated with pectinases. The pomace was gently dried at approx. 60 °C for 8.5 h in a H01 compact cabinet dryer (Harter GmbH, Stiefenhofen, Germany), giving a final moisture content of approx. 10 g/100 g. By means of a GM200 knife mill (Retsch GmbH, Haan, Germany), the dried pomace was comminuted without crushing the seeds. The seedless fibrerich fraction (skins, pulp, stems) was subsequently separated by automated sieving through a 0.6 mm sieve for 2 min at an amplitude of 1.4 mm (AS200, Retsch GmbH, Haan,

Germany). The moisture content of SBC was determined by oven drying at  $103 \pm 1$  °C to constant mass.

## **HTST** extrusion

Prior to extrusion, the seedless pomace was adjusted to a moisture content of 11 g/100 g, 16 g/100 g or 21 g/100 g with deionized water, and conditioned at 4 °C for at least 18 h. Extrusion was carried out in a DN 20 single screw extruder connected to an E330 DO-Corder (Brabender GmbH & Co. KG, Duisburg, Germany). Screw diameter was 20 mm, and die diameter was 2 mm. Feed screw speed was kept constant at 15 rpm and, based on preliminary experiments, the speed of the progressive screw n (compression ratio 1:2) was set to 150 rpm. A full factorial design was applied to investigate the impact of pomace moisture content (MC) and extrusion temperature T<sub>end</sub> on three levels each, giving nine test points per trial run (n=2). T<sub>end</sub> corresponds to the temperature set in the die zone (80 °C, 110 °C, or 140 °C). In the two trials, MC was randomised in the order of (a) 16/11/21 g/100 g and (b) 21/11/16 g/100 g, never starting with the lowest moisture to prevent clogging. Feed and barrel zone temperature were set to 80 °C each ( $T_{end} = 80$  °C), 80 °C and 110 °C, respectively ( $T_{end} = 110 \text{ °C}$ ), or 110 °C and 140 °C, respectively  $(T_{end} = 140 \text{ °C})$ . Average mass flow  $\dot{m}$  (kg/h) was calculated from the mass throughput  $m_e$  within 30 s in quadruplicate. The specific mechanical energy (SME [J/kg]) was derived by:

$$SME = \frac{P}{\dot{m}} = \frac{2\pi Mn}{\frac{m_e}{t}}$$
(1)

The energy input *P* was calculated from the mean values of torque (*M*) and screw speed (*n*), which were recorded every 15 s during extrusion and comprised of at least 15 recorded values per test condition, respectively. The expansion ratio, defined as radial volume increase of the extrudates, was calculated by dividing the average strand diameter (*n*=10) by the die diameter. Extrudates from each test point were collected, comminuted for 20 s in a knife mill and milled to below 500 µm using a ZM200 ultra-centrifugal mill (Retsch GmbH, Haan, Germany) to ensure defined particle size for further analyses. Extruded samples are encoded using temperature (°C)/pomace moisture (g/100 g) conditions, e.g. 80/11 refers to T<sub>end</sub>=80 °C and MC=11 g/100 g.

## **Planetary ball milling**

The SBC was micronized in a PM400 planetary ball mill (Retsch GmbH, Haan, Germany) at 300 rpm with direction reversal every minute. Each of the 250 mL grinding jars was loaded with 120 mL steel balls (d=3 mm) and 120 mL pomace (bulk volume). The temperature, monitored with an

infrared camera (Flir Systems AB, Sweden), increased to over 100 °C in preliminary experiments due to the lack of internal cooling devices. To prevent heating to over 60 °C in the main tests, the mill was stopped every 30 min. The jars were removed, immersed in liquid nitrogen, and milling was continued when the jars reached room temperature. After milling in duplicate for 2 h or 4 h, samples encoded as PM2 and PM4 were taken.

## Particle characteristics and colour

The particle size distribution of milled berry pomace was determined with a HELOS/KR-H2487 laser diffraction spectrometer (Sympatec GmbH, Clausthal-Zellerfeld, Germany). Prior to analysis at a dispersion pressure of 0.3 MPa, the powder was passed through a 1000  $\mu$ m sieve. Volume-based median values  $x_{50}$ ,  $x_{90}$  (90% of particles smaller than this size), Sauter mean diameter (SMD), and the specific surface area (SSA) referring to the monomodal spherical particles of SMD were calculated from the size distribution densities with the PAQXOS 4.3 evaluation software.

Colour was measured with a Luci 100 spectral colorimeter (D65 xenon lamp, 10° observer; Hach Lange GmbH, Düsseldorf, Germany). Pomace powders were filled in Quartz glass cylinders (d=34 mm) up to 4 mm height and colour primaries of triplicate measurements were transferred into the CIE-Lab colour space. The lightness L\*, hue angle h<sub>ab</sub> (describing the colour quality), chroma C\* (indicating saturation), and colour distance  $\Delta E^*$  to the initial SBC were calculated for interpretation [25].

## **Techno-functional properties**

The water solubility index (WSI) was determined according to Anderson et al. [26], and the water-binding capacity (WBC) was measured following Chen et al. [27]. Both methods were combined in one assay. Pomace powder (2.00 g) was mixed with 40 mL of deionized water and horizontally agitated for 60 min at 23 °C. The samples were then centrifuged at 7000g for 10 min. The supernatant was removed, weighed and freeze-dried (Beta 1-8 LSCbasic, Martin Christ Gefriertrocknungsanlagen GmbH, Osterode, Germany). The weight of the lyophilized supernatant was related to the dry mass of the pomace sample initially present in the tube and defined as WSI (g/100  $g_{DM}$ ). WBC was defined as the amount of water (g) that is bound per 1 g of dry pomace powder.

For determination of the swelling capacity (SWC), 0.20 g of berry pomace powder was weighed into a graduated tube. After adding 10 mL deionized water and mixing for 30 s using a vortex shaker, the tubes were placed in a rack for 18 h at room temperature. The volume of the swollen pomace powder was read from the graduation, and the SWC is

further expressed as mL per  $g_{DM}$ . Each powder of trial (a) and (b) was separately analysed twice for WSI, WBC, and SWC (n=4).

## Proximate composition and soluble carbohydrates

Fat content was analysed by acid hydrolysis and subsequent Soxhlet extraction with petroleum ether, crude protein using the Kjeldahl procedure (conversion factor 5.3) and ash content after incineration in a muffle furnace (4 h at 550 °C). SDF and IDF were analysed using a total dietary fibre enzyme kit (Megazyme u.c., Bray, Ireland) based on AOAC 991.43 [28] in duplicate.

D-Glucose and D-fructose content ( $c_{Glu}$ ,  $c_{Fru}$ ) were analysed in the freeze-dried supernatant after WSI determination based on AOAC 985.09 [28] using an enzymatic test kit (K-FRUGL; Megazyme u.c., Bray, Ireland). Absorbance was determined spectrophotometrically (Ultrospec8000; Biochrom Ltd., Cambridge, GB) at 340 nm.

## **Statistical analysis**

Analysis of variance (two-way ANOVA for extrudates, one-way ANOVA for micronized samples) with subsequent Tukey post hoc testing at  $p \le 0.05$  and regression analyses were conducted with SAS<sup>®</sup> OnDemand for Academics (SAS Institute Inc., Cary, USA). Before analysis, the technical replicates of trial (a) and (b) were pooled. Correlations are significant ( $p \le 0.05$ ) for coefficients  $r \ge 0.67$ .

## **Results and discussion**

## **Extrusion process indicators**

The seedless blackcurrant pomace was pressed into dense strands by extrusion. Only at  $T_{end} = 140$  °C, the strands were partially interrupted by small chunks, which indicated the onset of unsteady flow in the extruder barrel. By considering this effect, a higher extrusion temperature is not recommended for this material. Jing and Chi [13] found 130 °C as upper temperature limit for the processing of soy bean residues in a twin-screw extruder, because material puffed out unevenly and the screw showed clogging. Generally, the mass flow in this study was constant  $(2.05 \pm 0.16 \text{ kg/h})$  and not affected by  $T_{end}$ . Only at a moisture content of 21 g/100 g, a small but significant reduction to  $1.93 \pm 0.08 \text{ kg/h}$  was observed.

The extrudates showed an expansion ratio of  $1.02 \pm 0.01$ . This almost imperceptible expansion is typical for nonstarch carbohydrates. The dietary fibre-rich pomace is unable to include and stabilise gas bubbles in the matrix, so that the vaporised water leaks out instead [23]. As shown by



**Fig. 1** Specific mechanical energy during extrusion of seedless blackcurrant pomace at different temperature and varying moisture content. Lowercase letters indicate significant differences (p < 0.05) caused by pomace moisture; uppercase letters indicate significant differences caused by extrusion temperature (p < 0.05); the interaction of the parameters moisture and temperature was insignificant

various authors, the addition of 10% pomace already reduced the expansion of starch-containing extrudates significantly [22, 29, 30]. In case of extruded snacks containing different cereals, potato starch and blackcurrant pomace (28%) as main ingredients, expansion ratios between 2.3 and 4.9 were reached [31].

Figure 1 depicts the specific mechanical energy as a function of MC and T<sub>end</sub>. Both, low moisture (11 g/100 g) and low temperature (80 °C) were responsible for a significant increase in SME. Because screw speed was kept constant and mass flow rate was hardly affected, the SME correlated exclusively with the torque (r = 0.99, p < 0.001). A high screw torque is associated with a high viscosity of the material in the barrel zone which, in turn, is caused by a low initial moisture content of the material. The impact of moisture on the SME was repeatedly observed by different authors (e.g. [12, 15]) and, in case of lupin seed coats, these measures correlated significantly (p < 0.001; [14]). When moisture content was kept constant, Schmid et al. [12] found that the SME increased concomitantly with material temperature and complex viscosity of apple pomace, whereas the SME was independent of barrel temperature for lupin kernel fibre [15].

After extrusion, pomace moisture content was reduced from MC 11, 16 or 21 g/100 g to 5.6, 8.0 or 10.8 g/100 g, respectively. Hence, the samples lost approximately half of their initial moisture during extrusion. The final moisture content appeared to be independent from  $T_{end}$ .

#### Micronized pomace powders

Table 1 summarises particle size characteristics of seedless blackcurrant pomace before and after planetary ball milling.

**Table 1** Volume-based median value  $x_{50}$ ,  $x_{90}$ , Sauter mean diameter (SMD), and specific surface area (SSA) after planetary ball milling of seedless blackcurrant pomace

Milling time (h)	x <sub>50</sub> (µm)	x <sub>90</sub> (μm)	SMD (µm)	SSA (m. <sup>2</sup> /mL)
0	$256.5 \pm 2.1^{a}$	$532.2 \pm 16.9^{a}$	$76.2 \pm 0.7^{a}$	$0.1 \pm 0.0^{\circ}$
2	$7.0 \pm 0.1^{b}$	$22.5 \pm 00.3^{b}$	$3.6 \pm 0.0^{b}$	$1.7 \pm 0.0^{b}$
4	$4.0 \pm 0.1^{\circ}$	$10.7 \pm 00.1^{\circ}$	$2.5 \pm 0.1^{\circ}$	$2.5 \pm 0.1^{a}$

Mean values ( $\pm$  standard deviation) in a column with different superscripts differ significantly (p < 0.05)

Before milling, the cumulative particle size showed a broad sigmoid distribution reaching up to  $701 \pm 8 \ \mu m (x_{99})$  with only a few small particles  $(5.5\% < 30 \ \mu m)$ . The distribution was clearly narrowed during milling and 95.5% (PM2) respectively 99.8% (PM4) of the particles showed a size below 30–40  $\mu m$ , which corresponds to the human sensory detection threshold [32]. Particle size after 4 h milling was approximately half of that after 2 h milling. The respective median was 4  $\mu m$ , which indicates a 98% reduction of the initial particle size of SBC. Concurrently, the specific surface significantly increased from 0.1 m<sup>2</sup>/mL to 1.7 m<sup>2</sup>/mL and to 2.5 m<sup>2</sup>/mL for PM2 and PM4, respectively, which may indicate a higher adsorption capacity and a potentially improved bioavailability of nutrients and bioactive compounds [21].

In terms of particle reduction and time, the milling conditions chosen for blackcurrant pomace proved to be efficient when compared to planetary ball milling of other fruit and vegetable residues. For instance, okra particle size was reduced within 6 h by 91% from 162 µm to 15.2 µm ( $x_{50}$ ; [19]) and onion peels took 18 h for a 80% reduction from 770 to 153 µm ( $x_{50}$ ; [21]). Pulp-rich olive pomace was milled from 265 to 15.6 µm on average within 5 h (94% reduction). An olive stone-rich fraction was, however, more difficult to break and was characterised by coarser particles before and after ball milling [20]. It can be assumed that seeds in blackcurrant pomace would also impede particle size reduction, as they required higher impact in grinding experiments than cell wall components [7].

## Effects of thermo-mechanical treatments

#### Colour of processed pomace

Blackcurrant pomace appears dark red ( $L^* = 42$ ,  $C^* = 10.4$ ,  $h_{ab} = 29^\circ$ ; [7]) because of its anthocyanins, which account for approximately 85% of total polyphenols [33]. The separation of seeds intensified primarily the reddish colour as the chroma increased to 13.9 and the hue angle changed to 18° (Fig. 2). Extrusion of the pomace resulted in a colour distance  $\Delta E^*$  to the raw material of 2.7–4.9 (samples



**Fig. 2** Colour coordinates of seedless blackcurrant pomace (SBC) as affected by extrusion conditions (encoded with temperature  $[^{\circ}C]/$  pomace moisture [g/100 g]) or planetary ball milling for 2 h (PM2) or 4 h (PM4). Lightness L\* is proportional to circle area as indicated

80/16-140/21), and planetary ball milling caused a  $\Delta E^*$ of 7.9 and 8.7 for PM2 and PM4, respectively. Except for condition 140/21, the hue angle increased significantly after extrusion, to the greatest extent for MC = 11 g/100 g, but also when T<sub>end</sub> was low. The significant correlation between  $h_{ab}$  and SME (r=0.71) underlines that the colour changes are related to the shearing impact. The colour of the extruded powders with MC 11 g/100 g shifted to yellow, which can be explained by melanoidins formed through Maillard reactions. Furthermore, a loss in chroma, hence colour intensity, was observed with increasing T<sub>end</sub>. Similarly, the lightness decreased when moisture and extrusion temperature were high, most pronounced for 140/21 ( $L^* = 42$ ). In context of temperature treatments, polyphenols are prone to degradation. Short time (15 min) exposure to above 100 °C decreased total anthocyanins in dried currant pomace to a higher extent than 30 min below 100 °C, independent of pomace moisture [34]. In that study, however, the content of total polyphenols was similar after heat treatments between 80 °C and 120 °C, because more flavonols and hydroxycinnamic acids were detected at higher temperatures. A decrease in lightness was also observed for extruded apple pomace and lemon residues [11, 35] and slight browning occurred with high SME during citrus fibre extrusion [36].

In contrast to extrusion processing, the pomace became brighter through ball milling (up to  $L^*$  53.8 after 4 h). This can be attributed to the enlarged specific surface and

therefore changed light scattering, also found for milled onion peels [21]. The intensified chroma after 2 h milling was not observed after milling for 4 h. Because temperature increased up to ~55 °C during micronization, degradation of anthocyanins, hence the loss of red pigments, may have been initiated [33].

#### **Techno-functional properties**

The initial SBC was characterised by a swelling capacity of  $4.3 \pm 0.1 \text{ mL/g}_{DM}$ , a water-binding capacity of  $4.6 \pm 0.4 \text{ g/g}_{DM}$ , and a water solubility index of  $6.4 \pm 0.3 \text{ g}/100 \text{ g}_{DM}$ . After extrusion the SWC increased, on average and independent of temperature or moisture content, to  $5.0 \pm 0.2 \text{ mL/}$  $g_{DM}$  (Fig. 3, top). The micronized pomace showed enhanced swelling not only compared to the initial SBC, but also as compared to the extruded samples. After milling for 4 h, the SWC was  $7.6 \pm 0.2 \text{ mL/g}_{DM}$ . This increase may be taken as an indicator for a modified three-dimensional structure of the dietary fibre and for the presence of irregularly broken, porous cell walls [11, 13, 37]. Microscopic images of extruded insoluble apple fibre in water showed an immediate expansion up to 12-fold of the initial volume, which was also explained by an enhanced macromolecular porosity [12].

Although a porous structure facilitates the sorption of large amounts of water, the SWC does not necessarily correlate with the WBC. When the hydrated pomace was subjected to centrifugal force, it released more water after thermo-mechanical processing than the initial pomace (Fig. 3, middle). Solubilised pectin, which is expected to increase water binding, was probably largely destroyed through the pectinases used during juice processing. While the impact of T<sub>end</sub> was negligible, the WBC decreased significantly after extrusion with reduced MC (11 g/100 g), on average to  $3.3 \pm 0.2$  g/g<sub>DM</sub>. The WBC dropped to  $3.4 \pm 0.1$  g/ g<sub>DM</sub> after 2 h of planetary ball milling. The results indicate that cellulose fibrils are cleaved so that structural capillary forces are reduced [20, 36]. However, hemicelluloses can also be expected to be soluble after a mechanical breakdown of the fibre matrix [14]. Therefore, the WBC, which remained constant between 2 and 4 h milling, suggests overlapping effects as regards fibre matrix breakdown. The results substantiate the complex water-binding behaviour in mixed fibre systems, which are difficult to predict [38]. While the WBC of orange pomace increased [10], other citrus fibres entrapped less water after extrusion cooking [36].

Figure 3 (bottom) shows that the solubility of SBC increased after extrusion and primarily depended on moisture content before extrusion but not on extrusion temperature. The highest WSI  $(7.9 \pm 0.1 \text{ g/100 g}_{DM})$  was obtained for MC = 11 g/100 g and reflects an increase of 23.2% compared to the initial SBC. A positive correlation with SME (r=0.81) was found. Redgwell et al. [36] explained

Fig. 3 Techno-functional properties of seedless blackcurrant pomace (SBC) after extrusion at different temperature with varying moisture content (left) and after planetary ball milling (right). Horizontal planes in the left graphs indicate the level of the initial SBC. Lowercase letters indicate significant differences (p < 0.05) caused by pomace moisture; uppercase letters indicate significant differences caused extrusion temperature (p < 0.05); the interaction of the parameters moisture and temperature was insignificant. Different superscripts in the right graphs indicate significant differences (p < 0.05) between milling times



that high shearing forces promote the destruction of insoluble fibre and create fractions with lower molecular mass and enhanced hydrophilicity [21, 24]. The WSI of milled pomace increased gradually within the 4 h milling time up to  $7.6 \pm 0.0$  g/100 g. This solubility is substantial for food applications, which are based on emulsifying, foaming, or gelling. Micronized apple pomace for instance stabilised pickering emulsions due to its improved physico-chemical properties, such as small contact angle, higher solubility and increased water holding capacity [24]

#### Dietary fibre composition and soluble carbohydrates

The SBC comprised  $71.31 \pm 0.91$  g total dietary fibre (5.56 g SDF, 65.76 g IDF),  $10.42 \pm 0.04$  g protein,  $4.22 \pm 0.07$  g fat, and  $2.96 \pm 0.07$  g minerals per 100 g dry matter. The remaining difference of 11.08 g was defined as non-DF carbohydrates, which was confirmed by determination of  $7.28 \pm 0.41$  g fructose and  $3.96 \pm 0.11$  g glucose per 100 g in the freeze-dried extract obtained during WSI determination. As compared to whole blackcurrant pomace, the seedless

fraction comprised less fat (-79%) and protein (-34%), but 12 g/100 g (+21%) more total dietary fibre, of which 10.6 g were accountable to IDF [7].

The samples subjected to the highest mechanical impact (extruded at 80 °C with 11 g/100 g moisture, SME approx. 650 kJ/kg; 4 h planetary ball milling) were selected for comparison of dietary fibre composition. As shown in Table 2, neither the SDF nor the IDF content changed significantly during thermo-mechanical treatment. The IDF/SDF-ratio was in a magnitude of 12 for all powders, therefore far above the nutritionally and techno-functionally favourable ratio of 2:1 [39]. Probably, the mechanical impact was not sufficient for cleaving the hemicelluloses from the insoluble fibre matrix. Studies, who observed increased SDF after extrusion cooking, were performed with a predominantly higher SME (e.g. 1296 kJ/kg, [14]; 10,118 kJ/kg, [15]) than the maximum SME (649 kJ/kg) observed in this study. In contrast, an SME in the range of 365-479 kJ/kg was appropriate to increase the SDF of citrus fibre from 6 g/100 g to 23-36 g/100 g [36]. Jing and Chi [13] observed an overall increase of SDF after extrusion of soybean residues with a maximum at 115 °C. They accounted the loss at higher temperatures (130 °C) to burnt material and agglomerates formed through unsteady processing conditions.

Another tentative explanation for constant SDF but increased WSI can be assigned to oligosaccharides presumably formed during milling or extrusion but not determined by the dietary fibre method, which is based on solvent precipitation. Redgwell et al. [36] compared monosaccharide content in hydrolysed extracts after WSI determination with SDF extracts following AOAC 993.43 [28]. Although the overall fibre solubility was lower in WSI extracts, they contained 20 times more xylose and twice as much galactose and arabinose than the SDF extracts. In the studies of Huang and Ma [10] and Zhong et al. [14], the WSI correlated with SDF content.

The content of SDF also remained constant after planetary ball milling, and it is not completely clear which aspects prevented the degradation of IDF. Most planetary ball mills lack of integrated cooling systems [18]. Perhaps cooling of the grinding jars in between milling in this

Table 2Dietary fibre composition of seedless blackcurrant pomace(SBC), after extrusion at 80 °C with 11 g/100 g pomace moisture(E80/11) and after 4 h planetary ball milling (PM4)

	SDF (g/100 g <sub>DM</sub> )	IDF (g/100 g <sub>DM</sub> )	IDF/SDF
SBC	$5.56 \pm 0.17^{a}$	$65.76 \pm 0.74^{a}$	$11.84 \pm 0.38^{b}$
E80/11	$5.59 \pm 0.08^{a}$	$64.68 \pm 0.74^{a}$	$11.57 \pm 0.21^{b}$
PM4	$4.98 \pm 0.13^{a}$	$64.43 \pm 0.75^{a}$	$12.95 \pm 0.37^{a}$

Mean values (± half deviation range) in a column with different superscripts differ significantly (p < 0.05)

study prevented the conversion of dietary fibre. Although it was stated that an increased temperature favours crushing during planetary ball milling [18], high temperature was not a key factor for DF conversion during extrusion. However, authors who observed changes in DF after ball milling did not explicitly mention any cooling steps (e.g. [40, 41]). So it cannot be excluded that the temperature increase during milling had an effect in the aforementioned studies. Normally, an increase in SDF is accompanied by a decrease of IDF [19], which results in a more advantageous IDF/SDF ratio. Depending on the combination of ball size and rotational speed, the IDF/SDF ratio decreased for okara from 19 to 1.1 [19] and for olive pomace from 87 to 12 (stone-rich fraction) or from 6 to 3 (pulp-rich fraction; [20]). Both processed materials were characterized by an average particle size of 15 µm.

Figure 4 shows the glucose and fructose content of the freeze-dried pomace extracts. Overall, these sugars decreased after (thermo-)mechanical stress, most pronounced in extrudates with MC = 11 g/100 g, showing the highest SME. Again, the impact of extrusion temperature was not significant and neither was the difference between MC = 16 g/100 g and MC = 21 g/100 g. Fructose was significantly reduced after 2 h ball milling and glucose after 4 h. A negative correlation between  $c_{Glu}$  and the hue angle was found (r = -0.96), same as for  $c_{Fru}$  and the hue angle, which means that less sugars were present in yellow-brownish powders. Witczak et al. [23] also observed lower amounts of glucose and fructose after extrusion of blackcurrant pomace with corn flour, which was explained by the occurrence of Maillard reactions. In contrast, Huang and Ma [10] found more monosaccharides in extracts of extruded orange pomace and associated it with degraded cellulose and hemicelluloses. Micronized olive pomace showed no difference in glucose content compared to the un-milled reference [20].

Compared to extruded or micronized by-products in other studies (e.g. [11, 19].), blackcurrant pomace is rich in polyphenols [7]. Interactions of carbohydrate polymers with tannins were described previously, whereby the binding efficiency was increased by hydrophobic interactions [42]. Thus, low-methylated pectin showed higher binding affinity than high-methylated pectin, followed in decreasing order by xyloglucan, starch, and cellulose. Given that blackcurrant pectin is low-methylated [43], such interactions may explain the unchanged dietary fibre content and the lower release of monosaccharides. However, a lower amount of available sugars can be beneficial for lower energy intake during digestion and polyphenols were reported to counteract hyperglycaemic effects [3]. Different studies also reported on an increased antioxidant capacity after thermomechanical processing of fruit or vegetable pomace [11, 21, 24], and it may be concluded that extruded or micronized

Fig. 4 Sugar content of dried aqueous extracts of seedless blackcurrant pomace (SBC) after extrusion at different temperature with varying moisture content (left) and after planetary ball milling (right). Horizontal planes in the left graphs indicate the level of the initial SBC. Lowercase letters indicate significant differences (p < 0.05) caused by pomace moisture; uppercase letters indicate significant differences caused by extrusion temperature (p < 0.05): the interaction of the parameters moisture and temperature was insignificant. Different superscripts in the right graphs indicate significant differences (p < 0.05) between milling times



blackcurrant pomace presumably shows enhanced functional properties.

# Conclusion

Seedless blackcurrant pomace was successfully extruded without additives, which resulted in powders with increased swelling capacity, higher water solubility, and a lower water-binding capacity. Micronization proved to be a promising alternative for improved powder functionality. Constant dietary fibre content can be advantageous when it comes to food applications, since no processing losses are to be expected. Both thermo-mechanical treatments are promising to extend the applicability of fruit by-products, for instance as ingredient in beverages or fatbased systems. Micronized and hence more hydrophilic fibres may counteract sedimentation in liquids whereas an increased swelling capacity could have stabilizing effects on yoghurt-like systems. Prospective research should investigate the performance of modified pomace fibre in different food systems and analyse interactions with other compounds. Furthermore, the pomace can be treated with a higher mechanical impact, for instance using a twin-screw extruder or by high-pressure homogenization, to gain more knowledge on the degradation of the fibre matrix.

Acknowledgements The research project was carried out in the framework of the industrial collective research programme (IGF no. 20917 BG). It was supported by the Bundesministerium für Wirtschaft und Klimaschutz through the AiF (German Federation of Industrial Research Associations eV) based on a decision taken by the German Parliament. Thanks to Austria Juice GmbH for supplying the blackcurrant pomace and to Harter GmbH for giving support in pomace drying.

Author Contributions Conceptualization, H.R. and A.-M.R.; methodology and investigation, M.B. and A.-M.R.; writing—original draft preparation, A.-M.R.; writing—review and editing, H.R. and S.S.; supervision, H.R.; project administration, S.S. Authors Reißner, Brunner, Struck, and Rohm confirm that this is original work that is not submitted for publication elsewhere. All authors have approved the final version of the manuscript.

**Funding** Open Access funding enabled and organized by Projekt DEAL. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

#### Declarations

**Conflict of 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.

**Compliance with ethics requirements** This article does not contain any studies with human or animal subjects.

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