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Composition and physicochemical properties of dried berry pomace

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Abstract

BACKGROUND: Berry pomace is a valuable but still little used by-product from juice manufacture. When processed to a stable fruit powder, the composition differs from that of the whole fruit. To facilitate the application in foods, detailed knowledge of its composition and physicochemical properties is essential.

RESULTS: Blackcurrant, redcurrant, chokeberry, rowanberry and gooseberry were selected for analysis. All pomace powders had a high fibre content (>550 g kg^{-1}) and a fat content of up to 200 g kg^{-1}. Despite identical milling conditions, the particle sizes of the pomace powders varied. This can be traced back to seed content and brittleness, becoming also apparent in surface characteristics. Blackcurrant pomace powder differed from other varieties by its low water binding capacity (3.2 g g^{-1}) and a moderate moisture uptake, whereas chokeberry pomace powder showed the highest polyphenol content and rowanberry pomace powder was rich in flavonols.

CONCLUSION: The results obtained in this study give a comprehensive overview on the properties of berry pomace powder and allow drawing conclusions on their applicability for being used in complex food systems.

INTRODUCTION

The recommended daily dietary fibre intake of human adults is 25 g, and 70–80% of that amount should be insoluble.[1,2] One prominent possibility to increase fibre content of the diet for enhancing the still-too-low intake is to supplement foods with fibre from cereal origin.[3] Alternatively, increased fibre intake may be achieved by adding fruit processing by-products. A further use of, for instance, pomace adds

value to a material that is usually treated as waste, and therefore contributes to an improved sustainability of the agri-food processing chain.

The composition of apple and citrus pomace powders has been widely studied, and respective fibre products are commercially available.[2,4] These pomaces are especially interesting because of their high pectin content, but recent research also examined applications of apple and citrus fibre that are based on their chemical and technofunctional properties.[5,6] Apart from dietary fibre, berry pressing residues contain high amounts of bioactive compounds which may trigger health promoting effects once incorporated in the human diet,[7] and therefore make them an interesting study subject.

Water soluble antioxidant components located in the cell vacuole (for instance, chlorogenic acid) are released into the juice during pressing whereas compounds with low solubility (e.g., anthocyanins) that are associated with the cell wall remain in the pomace.[8] It has been shown that the total dry matter (d.m.) related polyphenol content of chokeberry and blackcurrant press residues is 1.3-1.7 times higher than that of the respective berries.[9,10] Physiological functions of dietary fibre in human digestion derive from their physicochemical properties, including water and fat binding capacity, swelling ability, and rheological properties.[11] In addition, quantification of the respective properties allows drawing conclusions on possible food applications. For instance, fibre with a high fat binding capacity might be best applied to stabilize fat in emulsion based products, whereas high water binding can be linked to decreasing syneresis in hydrogels, or altering food viscosity and texture.[4,12]

Although it has been mentioned that pomace from pooled lots of berries from different producers may contain a large number of different pesticides,[13] the

few studies available on the topic indicated that berries usually carry a low pesticide level, and that the pesticide level of pressing residues does not exceed that of fresh fruits.[13,14,15] By taking these facts and the positive consumer image into account, by-products of berry juice production may serve as promising food ingredients with health promoting effects.[7,16] Once processed into stable powder, a wide range of applications is possible. Prominent examples are baked or extruded cereal products where dried pomace may be used for partially replacing wheat, fat or sugar,[17-19] or in meat or fish products for enhancing texture, and water and fat binding properties.[12,20]

In industrial juice production berries are usually crushed or mashed, heated to 40-50 °C and treated with depectinising enzymes to disrupt the viscous gels formed during mashing so that extraction is facilitated.[7,21] The remaining pomace is, because of its sugar content, highly susceptible to microbial spoilage[22] so that immediate drying, accompanied by optimum milling, is necessary to control the fate of polyphenols and other heat-sensitive compounds.[8]

Fortifying foods with ingredients with high fibre content often (e.g.[12,17-20]) results in an unfortunate alteration of product characteristics which, to some extent, may be counteracted by formulation or process adaptation.[23] Knowledge of technofunctional properties in combination with physical characteristics and chemical composition is required to evaluate the potential for the application of dried berry pomace. The aim of the present study was to investigate composition, microbiological state, polyphenol content and physical properties of processed berry pomace, and to show similarities and differences among five different berry varieties. The information on the characteristics of berry pomace powder is helpful to facilitate its further application in foods.

MATERIALS AND METHODS

Berry pomace as raw material and processing of pomace powder

Berry pomace was collected from juice processors in the 2015 harvesting period on the day of pressing. Processed berry varieties were *Ribes nigrum* blackcurrant (BC, from Döhler GmbH, Neuenkirchen Hadeln, Germany), *Aronia melanocarpa* chokeberry (CB), *R. rubrum* redcurrant (RC) and *R. uva-crispa* gooseberry (GB) from Kelterei Walther GmbH, Arnsdorf, Germany, and *Sorbus aucuparia* rowanberry (RB, from Kelterei Kühne, Haselbachtal, Germany). The pomaces contained stems, seeds and skins and were stored at -20 °C in ziplock bags in 10–15 kg batches.

After thawing for 24 h at 8 °C berry pomace was dried in an ULE 400 convection oven (Memmert GmbH & Co. KG, Schwabach, Germany) at 60 °C for 24 h, sufficient to reach a moisture content below 60 g kg^{-1}. The dried material was subsequently milled in a ZM 100 ultra-centrifugal mill, equipped with a 0.5 mm sieve (Retsch GmbH, Haan, Germany) at 14,000 rpm, and used for all subsequent analysis except for seed – and polyphenols content.

For polyphenol analysis, fresh pomace was freeze-dried at -42 °C for 72 h in an Alpha 1-2 LDplus (Martin Christ Gefriertrocknungsanlagen GmbH, Osterode, Germany) and milled after lyophilisation, again by using the ZM 100 mill operating at conditions described above.

Microbiological analysis

Five grams of fresh berry pomace or dried berry pomace powder were suspended in 45 mL sterile peptone water (1.0 g L^{-1} casein peptone, 8.5 g L^{-1} sodium chloride) and homogenized in a Stomacher 400 (Seward Ltd, Worthing, UK) for 5 min. The

solution was decimally diluted and pour-plated with plate count agar (incubation: 30 °C, 48 h) for total mesophilic count and yeast extract glucose chloramphenicol agar (incubation: 25 °C, 72 h; both from Carl Roth GmbH & Co. KG, Karlsruhe, Germany) for yeast and moulds.

Proximate analysis

Moisture content of fresh and dried berry pomace was determined by drying at 103 °C to constant mass. Fat content was analysed by acid hydrolysis and subsequent Soxhlet extraction with petroleum ether, crude protein by the Kjeldahl procedure (conversion factor 5.3), and ash content after incineration in a muffle furnace (4 h, 550 °C). Soluble (SDF) and insoluble dietary fibre (IDF) were analysed using the total dietary fibre kit (Megazyme u.c., Bray, Ireland) based on AOAC 991.43.[24] The remaining difference to 100% was considered as being non-DF carbohydrates. Total acidity was determined as tartaric acid equivalent after titration of an aqueous pomace powder suspension (50 g L^{-1}) with 0.1 mol L^{-1} sodium hydroxide.

Extraction and HPLC-DAD-MS analysis of phenolic compounds

Pomace samples were extracted using a pressurized hot water extraction method.[25] Briefly, 1 g of milled berry pomace was extracted with degassed Milli-Q water containing 50 mL L^{-1} ethanol and 10 mL L^{-1} formic acid in a 10.0 mL extraction cell using an ASE-350 system (DIONEX Softron GmbH, Germering, Germany). Extractions were performed at 99 °C with 5 min preheating and 1 min static extraction. After that, the sample was flushed with fresh solvent (50% of the vessel volume). Extracts were diluted to a final volume of 25 mL with the extraction liquid

(see above) and stored at -20 °C. Prior to analysis, the extracts were centrifuged for 5 min to remove any undissolved particles.

HPLC-DAD analyses were performed on a Dionex UltiMate-3000 HPLC system equipped with an online degasser, a dual gradient solvent pump, a thermostatted autosampler, a column oven, and a Diode Array Detector (DAD). The system was controlled and data acquired using Chromeleon 6.80 software. Separations were performed by injecting 5 µL of the extract on a porous-shell fused core Ascentis Express C_{18} column (100 mm × 3.0 mm, 2.7 µm; Supelco, Bellefonte, PA). Column temperature and flow rate were set to 45 °C and 350 µL min^{-1}, respectively. The mobile phase consisted of (A) water and (B) methanol, both containing 10 mL L^{-1} formic acid. The gradient starting with 5% (B) was kept constant for three min and then linearly increased to 80% (B) during 30 min. Chromatograms were recorded at 280 nm for total phenolics, at 350 nm for flavonoids and at 520 nm for anthocyanins. The mass spectrometer was scanning from 50 to 1100 m/z, the cone voltage was set to 35 V and the capillary voltage to 2.5 and 3.0 kV for positive and negative ESI mode, respectively. The desolvation gas flow rate was 800 L h^{-1} at a temperature of 550 °C and the cone gas flow rate was 40 L h^{-1}. The source temperature was 120 °C. MS^E was performed with collision energy ramped from 15 to 50 eV. The system was controlled by and data acquired using MassLynx 4.1 (Waters MS Technologies, Sollentuna, Sweden).

Determination of physical properties

Particle size

Particle size distribution of milled berry pomace was determined with a HELOS/KR-H2487 laser diffraction spectrometer (Sympatec GmbH, Clausthal-Zellerfeld,

Germany). Before analysis at a dispersion pressure of 0.3 MPa the powder was passed through a 2000 µm sieve. Volume based median values x_{50} and x_{90}, and specific surface area was calculated from the size distribution densities.

Pomace seed content

Dried pomace was ground for 3 min at maximum speed (level 10) in a berlinett CM5100 knife mill (VEB EMK, Berlin, Germany). It was verified in preliminary experiments using light microscopy that, under these conditions, only stems and skins were comminuted whereas seeds remained intact. Subsequently, the seeds were separated using an AS 200 vibratory sieve shaker (Retsch GmbH, Haan, Germany), weighed, and expressed as fraction of whole dried pomace.

Microstructure

Pomace powder was vacuum coated with platinum and observed under a field emission scanning electron microscope (model Ultra 55 FESEM, Zeiss, Oberkochen, Germany). Each sample was analysed in duplicate.

A light microscope (Nikon Eclipse 80i, Nikon Co., Ltd., Tokyo, Japan) was used to study the structure of different pomaces according to Hernández-Carrión et al.[26] The autofluorescence of the samples containing phenolic compounds was observed while using a mercury arc lamp with a TRITC filter (λ_{ex}=543/22 nm, λ_{em}=593/40 nm) as excitation source. Samples were placed on a microscope slide, covered with a cover slip and observed at 200x magnification. The images were captured and stored at 1280 x 1024 pixels using the microscope software (NIS-Elements F, Version 4.0, Nikon, Tokyo, Japan).

Colour

Colour of berry pomace powder was measured in the CIE-Lab colour space with a Luci 100 spectral colorimeter (D65 Xenon lamp, 10° observer; Hach Lange, Düsseldorf, Germany). Lightness L*, hue angle h_{ab} and chroma C* were calculated from the colour primaries and used for further interpretation.[27]

Determination of technofunctional properties

Water activity and sorption isotherms

Equilibrium water activity a_W of the pomace powders was measured with a LabMaster-aw benchtop instrument (Novasina AG, Lachen, Switzerland). A Q5000 SA dynamic vapour sorption analyser (TA Instruments, Eschborn, Germany) was used to analyse moisture sorption and desorption at 20 °C. After loading approx. 5 mg sample, relative humidity RH was kept at 0% until mass change was less than 0.001% within 5 min. RH was then increased to 90% in 10% steps and, finally, to 95%; at each level, mass was continuously recorded until no further change was observed (<0.01% for 5 min), and desorption isotherms were recorded similarly.

The Guggenheim-Anderson-DeBoer (GAB) model was applied for fitting isotherms, with *x* being the moisture load (g g^{-1}) at a particular RH (= 100 a_W), x_0 being the monolayer moisture load, and *C* and *k* referring to the monolayer and multilayer energy constants, respectively:[28]

$$x = \frac{x_0\ C\ k\ a_W}{(1 - k\ a_W)(1 - k\ a_W + C\ k\ a_W)}$$

Water- and oil binding capacity

Water binding capacity (WBC) was determined according to Zahn *et al.* (2013).[29] One gram pomace powder was mixed with 30 mL deionised water, vigorously

agitated, and kept at 20 °C for 30 min. The samples were centrifuged at 2000x*g* for 10 min. The supernatant was removed, weighed and subjected to dry matter determination (oven method). Initial powder moisture was considered in the calculation of water and dry matter content of the remaining sediment. WBC is defined as g water that is bound per g dry pomace powder under these conditions. For swelling capacity determination, 0.2 g berry pomace powder was weighed into a graduated tube. After adding 10 mL deionised water and mixing, 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 swelling capacity is further expressed as mL per g d.m.[30]

To determine the potential to absorb fat, 0.5 g dried pomace was mixed with 10 mL canola oil and kept for 18 h at room temperature. The sample was centrifuged at 10,000x*g* for 30 min, the supernatant discarded and the mass of the pellet determined. In contrast to WBC measurements, where particles remaining in the supernatant were taken into account, the powder particles suspended in oil sedimented completely during centrifugation. Oil absorption capacity (OAC) is defined as g oil per g dried pomace powder.[31]

Statistical analysis

All experiments were carried out in triplicate except dietary fibre content determination (n=4), and total acidity and moisture sorption (n=2). Analysis of variance with subsequent Student-Newman-Keuls post hoc-testing at $P \leq 0.05$ was conducted with SAS University Edition 6p.2 (SAS Institute Inc., Cary, USA).

RESULTS AND DISCUSSION

Composition of pomace and pomace powders

The moisture content of fresh pomace ranged from 516.0–764.8 g kg^{-1} (Table 1), and was related to processing conditions. Vagiri & Jensen[32] reported that it is especially thermal and enzymatic degradation of pectin that affect juice yield and the distribution of cell compounds between juice and pomace. The companies that manufactured juice from BC, RC and CB treated the respective mashes with pectinase at 50 °C before pressing. Rowanberry and gooseberry mashes were pressed at room temperature without prior enzymatic treatment so that moisture content was significantly higher (approx. 750 g kg^{-1}).

Moisture content of dried pomace, containing crushed skins, seeds and stems, was below approx. 50 g kg^{-1}. The fat content ranged from 36.1-202.1 g kg^{-1} d.m. and was highest for BC pomace. Pomace fat content largely depends on its seed content;[33] the relative amount of seeds in the pomace was highest for blackcurrant and redcurrant pomace (61% and 40%, respectively), and lowest for chokeberry and rowanberry (20% and 8.4%, respectively). This is comparable to Sójka *et al.*[34] who reported 21.1% seeds in CB pomace and 57.1 – 62.1% in BC pomace. Protein content ranged between 59.7 g kg^{-1} for chokeberry, and 157.1 g kg^{-1} for blackcurrant pomace. Literature data are scarce but are, in case of BC, given by 130 g kg^{-1} pomace.[33,35]

The content of insoluble dietary fibre varied between approx. 500–600 g kg^{-1} d.m., and that of SDF between approx. 40–70 g kg^{-1}. As published by the Finnish National Institute for Health and Welfare,[36] total fibre content of fresh berries increases in the order of GB<RC<BC<RB, which is in accordance with the pomace results (Table 1). The highest total dietary fibre content is evident for rowanberries (approx. 660 g kg^{-1}), which might be caused by some remaining pectin due to the

lack of enzymatic mash treatment before pressing. The fact that the IDF content of skin powder from blackcurrant and chokeberry pomace was specified as being 601 and 666 g kg^{-1} dry matter, respectively,[37] indicates that skins can be considered as the main fibre sources in pomace.

The acidity of dried pomace was, expressed as tartaric acid equivalents, highest for gooseberry (4.1 g kg^{-1}), and lowest for chokeberry (1.1 g kg^{-1}), and pH of the respective suspensions ranged from 3.32 (GB) to approx. 4.5 (BC). The fact that organic acids are transferred into the juice can be taken as explanation for the low acidity of pomace with low moisture (i.e., BC and CB). Residual carbohydrate concentration ranged from 22.0-288.8 g kg^{-1} d.m. The observed differences can be attributed to berry composition, the effectiveness of juice extraction, but also to low molecular mass fibre and trace amounts of starch that were not determined as SDF or IDF.

Fresh berry pomace is highly susceptible to microbiological spoilage, and immediate processing is necessary for further use. Total aerobic viable count in fresh pomace ranged from $1.2x10^3$–$1.0x10^6$ cfu g^{-1}, and the yeast and mould count from $1.0x10^2$–$2.0x10^5$ cfu g^{-1} (Table 2). The large differences can be attributed to different contamination levels, but also to different treatments during harvesting, transport and pressing. Since the bacterial count does not exceed $1.0x10^7$ cfu g^{-1}, the standard value of the German Society for Hygiene and Microbiology for cut fruits, all pomace samples can be considered as microbiologically safe.[38] The recommended limit for yeasts and moulds of $1.0x10^5$ cfu g^{-1} was, however, exceeded in fresh BC and CB pomace. The time/temperature profile applied during convective drying (60 °C for 24 h) decreased the yeasts and moulds count in all dried pomaces to below the limit of $1.0x10^4$ cfu g^{-1}.[38] Depending on fruit variety

and initial moisture content, total viable count either decreased or increased by approx. one magnitude. For comparison, the acceptable viable count limit for dried sauces is given by $1.0x10^6$ cfu g^{-1}.[38] Both product acidity and water activity (highest a_W was 0.251 for RC; Table 3) imply that there is a low risk of microbiological and enzymatic activity,[39,40] and that the powders can be considered as stable.

The total polyphenol content can be analysed by relatively simple methods such as the Folin Ciocalteau assay,[41] or by considering peak areas identified by HPLC-DAD.[42] In this study, we combined qualitative analysis of MS with total peak areas obtained by DAD. Supporting Information shows representative chromatograms of the extracts at the respective wavelengths (Figure S1). In general, the chromatographic profiles obtained in this study are in agreement with literature data for chokeberry,[43] redcurrant and blackcurrant,[44] gooseberry[45] and rowanberry.[46] Tentative identification of the individual compounds (Table S1) was based on a combination of the obtained exact mass (*m/z*), MS fragmentation pattern and the retention time of the respective peaks.

As shown in Figure 1, chokeberry has the highest total phenolic content as measured at 280 nm, which is because of the high content of anthocyanins. The anthocyanin content is approx. five times higher than that of blackcurrant pomace, which showed the second-highest values. Compounds detected at 520 nm (Figure 1S: peaks 11, 12, 15 and 29) were identified as cyanidin 3-galactoside, cyanidin 3-glucoside, cyanidin 3-arabinoside and cyanidin 3-xyloside (Table S1).[47,48] Chlorogenic acid and chlorogenic acid derivates (*m/z* 353) were detected in high intensities (peaks 1 and 5 in Figure S1). At 520 nm, nine peaks were detected in blackcurrant extract compared to four peaks in redcurrant which explains the intense colour of blackcurrant. Free and glycoside forms of quercetin and

kaempferol (Table S1) are the main flavonoids detected in blackcurrant and redcurrant.[44,49] Gooseberry has a variety of flavonoids and anthocyanins; however, their abundance is low compared to other berries (see total peak area at 280 nm). Although the colour of gooseberry is orange, ten peaks were detected at 520 nm representing anthocyanins and confirmed by the exact *m/z* and previous studies.[45] In contrast, in rowanberry one peak only was detected at 520 nm with *m/z* 449, identified as cyanidin-3-galactoside. On the other hand, rowanberry is rich in chlorogenic acid and its derivates (peaks 1, 5, 6 and 33). Proanthocyanins in dimeric or trimeric form generally show low peak intensities. To overcome this drawback, ion chromatograms were used to detect this group of compounds using the exact masses of *m/z* 577.134 and 865.198 for dimers and trimers, respectively. Few peaks (peaks 4, 26, 30 and 41) where detected at low intensity compared to flavonoids and anthocyanins.

Physical and technofunctional properties

Despite similar milling conditions, the particle size median differed significantly (Table 3) with x_{50} values ranging between 86.4 µm (CB) and 112.7 µm (BC). CB and RB are Rosaceae whereas BC, RC and GB of the Grossulariaceae genus *Ribes* are botanically classified as berries. It can be presumed that *Ribes* seeds are less susceptible to fracture so that higher stresses are needed to obtain a comparable particle size. With regard to the x_{90} percentile, between-variety differences were less prominent.

Colour of the pomace powders ranged from dark purple (CB) to dark yellow (RB), depending on berry colour (Figure 2). It is the anthocyanins with their antioxidant activity that are mainly responsible for the colour of berries.[50] In

blackcurrant, for example, the main anthocyanins are delphinidin-3-glucoside, delphinidin-3-rutinoside, cyanidine-3-glucoside and cyanidin-3-rutinoside.[9] The hue angle was in the red domain for CB (h_{ab}=20.1°) and increased up to h_{ab}=69.2° for RB pomace; colour saturation ranged from C*=8.3 (CB) to 31.5 (RB).

As can be seen from the FESEM pictures (Figure 3), the pomace powders RC and GB contain particles that still show the cellular structure of the original fruit. Specifically, in RC pomace powder particles, even a high degree of cellular turgor was observed, which could be related to the significantly higher moisture content. On the other hand, the particles of RB and CB, which had a low moisture content, appeared unstructured without exhibiting a well-defined cellular structure. The BC particles had an intermediate structural integrity; the presence of cells or cellular structures can be hardly observed in this powder.

In addition, the BC particles showed a cohesive and fused network structure. This structure is in line with compositional results since this pomace powder had the highest protein and fat content. Thus, the presence of protein seems to favour the structural network organization of the particles, while the fat would be acting as a binder that prevents fracture of the particles into smaller pieces. This could be the reason for their significantly larger size if compared to the other pomace powders, as it is summarized in Table 3. On the other hand, the pomace powder particles of RB and CB did not show a network structure but seemed to be built by small and unstructured pieces, superimposed one on the other. This lack of cohesive structure can be related to the significantly lower contents of protein and fat that were evident for these pomace powders.

Bright field microscopic images of BC powder pomace (Figure 4, left) showed cohesive particles forming a network structure, as it was also observed by

FESEM. Small particles seem to be joined intimately to form larger particles. However, in CB pomace powder, small isolated particles of different shape and size are evident, with the reddish coloration being due to their content in anthocyanidins. Although information is scarce, it is known that the derivatives of the cyanidins are fluorescent.[51] In this sense, it was observed that CB pomace powder (Figure 4, right) exhibited a higher fluorescence intensity than BC pomace powder. This is in agreement with the results obtained for polyphenol content (Figure 1), which showed significantly higher contents for CB pomace powder.

Blackcurrant pomace exhibited the lowest water binding capacity (3.2 g g^{-1} d.m.), most likely because of degraded pectin and high fat content,[52] and similar water binding capacities are reported in the literature.[53] RB and GB that were not enzymatically treated before pressing showed significantly higher WBCs (approx. 4.7 g g^{-1}). Furthermore, the hydration properties of fibres with a high content of primary cell wall components (hemicellulose, pectin) are enhanced compared to fibre with a high content of secondary cell wall components (cellulose, lignin) that are mainly present in seeds and stems.[54] According to Rosell *et al.*[55] a low particle size is responsible for low WBC, which is true for chokeberry with its low water binding capacity (3.85 g g^{-1} d.m.). In contrast larger surface areas favour water adsorption, as can be seen from the comparatively high swelling capacity of this variety (6.7 ml g^{-1} d.m.). Swelling capacity of other pomace powders decreased in the same order as WBC. Oil absorption capacity is a measure of oil retention in foods, and rather depends on particle porosity than on chemical composition or molecular affinity to oil.[52,56] Among the pomace varieties OAC did not differ significantly but ranged from 1.91-2.27 g g^{-1} d.m., a similar range as observed for pineapple pomace (2.01 g g^{-1}).[57] For comparison, wheat flour (0.85 g g^{-1}) and citrus

fibres (1.2 – 1.8 g g^{-1}) show lower, mango peels and apple fibres higher oil absorption capacities (2.7 and 3.36 g g^{-1}, respectively).[58,59]

As stated by Chen *et al.* [60] the WBC, determined by the centrifugation method, is appropriate to characterise foods with a_W >0.98 but sorption isotherms are recommended as source of supplementary information concerning water retention. Moisture adsorption and desorption isotherms (Figure 5) show a moderate uptake of moisture for up to 60% RH, resulting in respective equilibrium moisture loads of below 100 g kg^{-1}. It can be assumed that chemisorption plays a minor role whereas capillary and swelling effects, that mean moisture binding in small and subsequently in larger pores, predominate. The pronounced increase of moisture adsorption above 80% RH can be attributed to sugar crystals that start to dissolve at this humidity. Differences in moisture uptake between berry varieties become pronounced at high relative humidity. However, the impact of particle size of the powders is less prominent as can be seen from black currant (Supporting information, Figure S2). Doubling the x_{50} (112.7 µm to 243.2 µm) and x_{90} (271.3 µm to 563.3 µm) showed no significant differences in moisture adsorption below 30% RH and above 80% RH.

The application of mathematical models to isotherms may help detecting details about interactions between moisture and sorbent. GAB modelling resulted in R^2>0.997 for adsorption (A) and >0.992 for desorption (D), with excellent approximation up to 90% RH (Table 4). Except for BC (Typ II, C k>2), the adsorption follows Typ III isotherms (C k<2) of Brunauer's classification while all desorption curves showed sigmoidal shape.[61] The hysteresis areas indicate that, for drying processes, either lower RH or higher temperatures are essential to gain comparable residual moisture. Moisture load at monolayer adsorption x_0 was lowest

for BC (42.29 g kg^{-1}) and highest for CB (65.74 g kg^{-1}) which can be linked to volume-based specific surface areas that increased in the same order (Table 3). Differences between A and D isotherms become apparent for the monolayer enthalpy constant *C* (monolayer binding forces during desorption are many times higher than during adsorption; GB 3.7 fold, RC 17 fold), whereas variations of berry variety are negligible. The transition of mono- to multilayer adsorption is unincisive (except for BC), which becomes apparent from the low *C* and the absent bend of the sorption isotherms at 10% RH. *k* which points on multilayer interactions between moisture and sorbent is lower during adsorption, indicating weaker binding forces. BC shows however an inverse behaviour, with *k* differing significantly from those of other powders. Besides surface characteristics (less porous surface area), a reduced moisture sorption can be linked to low carbohydrate contents. As described by Witczak *et al.* [62] sugar fortified orange peel showed a sharp increase of moisture uptake above 60% RH, and significantly more moisture was adsorbed than by dried peels. WBC of fruits is mainly determined by sugar content and surface characteristics, whereby it turned out that convective drying resulted in minor porosity and hygroscopicity than other drying methods.[63] Compared to other fruits and their by-products, powders from berry pomace adsorb less moisture at 20/25 °C, due to processing and compositional properties.[62,64,65] As regards presumptive applications of dried berry pomace as food ingredient, a wide range of food systems is possible, particularly sweet or savoury baked products. Depending on berry variety and substitution level, the powders will have an impact on colour, rheological properties, texture and nutritional value.[17,18,66]

CONCLUSIONS

Drying and subsequent milling of berry pomace resulted in colour-intense fruit powders with considerable amounts of polyphenols. Pomaces of *Ribes* genus (BC, RC, GB) were found to contain more protein, and higher amounts of seeds and therefore fat compared to chokeberry and rowanberry. These properties are important for interpreting differences in particle size. As observed by FESEM and laser diffraction, powder particles with a less intact cellular structure showed high specific surface areas. Moisture sorption was dominated by capillary effects, but was also influenced by the content of carbohydrates. As particle size seems to have a significant impact on hydration properties, further studies that show how processing conditions influence pomace powder characteristics would be valuable.

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Table 1. Proximate composition of berry pomace powder.

Parameter	Blackcurrant	Redcurrant	Gooseberry	Rowanberry	Chokeberry
Pomace moisture (g kg^{-1})	516.0 ± 0.8^{e}	621.5 ± 6.9^{c}	748.2 ± 8.3^{b}	764.8 ± 7.2^{a}	549.8 ± 3.2^{d}
Powder moisture (g kg^{-1})	34.3 ± 0.1^{c}	51.8 ± 0.2^{a}	50.5 ± 0.4^{b}	26.9 ± 0.1^{d}	27.2 ± 0.2^{d}
Fat (g kg^{-1})*	202.1 ± 2.2^{a}	142.3 ± 3.5^{b}	109.3 ± 0.2^{c}	39.7 ± 1.3^{d}	36.1 ± 1.1^{d}
Protein (g kg^{-1})*	157.1 ± 0.6^{a}	117.6 ± 2.7^{c}	124.0 ± 0.4^{b}	70.9 ± 0.8^{d}	59.7 ± 1.1^{e}
Ash (g kg^{-1})*	26.6 ± 0.3^{d}	30.0 ± 0.6^{b}	34.0 ± 0.2^{a}	28.4 ± 0.6^{c}	19.2 ± 0.0^{e}
SDF (g kg^{-1})*	39.7 ± 2.9^{b}	70.0 ± 2.9^{a}	70.4 ± 3.7^{a}	76.8 ± 7.3^{a}	70.4 ± 13.5^{a}
IDF (g kg^{-1})*	551.6 ± 16.5^{b}	510.8 ± 8.1^{cd}	495.6 ± 9.4^{d}	594.9 ± 15.3^{a}	524.6 ± 11.0^{c}
Titratable acid (g kg^{-1})*	0.9 ± 0.0^{e}	2.8 ± 0.1^{b}	4.1 ± 0.1^{a}	2.3 ± 0.0^{c}	1.2 ± 0.0^{d}
Carbohydrates (g kg^{-1})*	22.0	126.5	162.6	187.1	288.8

Mean values (± standard deviation, n=3) in a row with different superscripts differ significantly (P<0.05). *, dry matter related content. SDF – soluble dietary fibre, IDF – insoluble dietary fibre

Table 2. Microbial counts of fresh and dried berry pomace.

Berry pomace variety	Total viable count (cfu g^{-1})		Yeasts and moulds (cfu g^{-1})	
	Fresh pomace	Dried pomace	Fresh pomace	Dried pomace
Blackcurrant	$1.0 \cdot 10^6$	$1.6 \cdot 10^3$	$1.7 \cdot 10^5$	$1.2 \cdot 10^3$
Redcurrant	$1.2 \cdot 10^3$	$6.1 \cdot 10^4$	$2.3 \cdot 10^2$	$2.9 \cdot 10^2$
Gooseberry	$1.3 \cdot 10^3$	$8.8 \cdot 10^4$	$1.0 \cdot 10^2$	$1.0 \cdot 10^2$
Rowanberry	$2.0 \cdot 10^5$	$6.3 \cdot 10^5$	$1.1 \cdot 10^2$	$1.0 \cdot 10^2$
Chokeberry	$2.0 \cdot 10^5$	$2.1 \cdot 10^4$	$2.0 \cdot 10^5$	$2.3 \cdot 10^2$

Table 3. Physical and technofunctional properties of berry pomace powder.

Parameter	Blackcurrant	Redcurrant	Gooseberry	Rowanberry	Chokeberry
Relative seed content (%)	61.0 ± 1.0^{a}	40.4 ± 5.3^{b}	34.2 ± 1.4^{c}	8.4 ± 0.2^{e}	22.1 ± 0.1^{d}
Particle size (µm)					
x_{50}	112.7 ± 2.4^{a}	107.2 ± 2.6^{b}	095.4 ± 2.9^{c}	095.1 ± 1.6^{c}	86.4 ± 1.1^{d}
x_{90}	271.3 ± 9.9^{a}	262.7 ± 14.4^{a}	244.1 ± 2.6^{b}	263.1 ± 5.3^{a}	239.9 ± 2.5^{b}
Specific surface area (m^2 mL)	0.08 ± 0.01	0.09 ± 0.00	0.14 ± 0.04	0.24 ± 0.00	0.28 ± 0.01
Water activity (-)	0.100 ± 0.001^{d}	0.251 ± 0.001^{a}	0.169 ± 0.001^{b}	0.112 ± 0.002^{c}	0.091 ± 0.001^{e}
Water binding capacity (g g^{-1} dry matter)	03.20 ± 0.20^{c}	04.06 ± 0.08^{b}	04.65 ± 0.18^{a}	04.74 ± 0.61^{a}	3.85 ± 0.06^{b}
Swelling capacity (mL g^{-1} dry matter)	05.50 ± 0.15^{c}	06.12 ± 0.39^{bc}	06.14 ± 0.17^{bc}	07.09 ± 0.19^{a}	6.70 ± 0.43^{ab}
Fat absorption capacity (g g^{-1} dry matter)	02.00 ± 0.09^{a}	01.94 ± 0.15^{a}	02.06 ± 0.17^{a}	02.27 ± 0.13^{a}	1.91 ± 0.12^{a}

Mean values (± standard deviation, n=3) in a row with different superscripts differ significantly (P<0.05).

Table 4. GAB Coefficients of fitted sorption isotherms of berry pomace powder.

Parameter		Blackcurrant	Redcurrant	Gooseberry	Rowanberry	Chokeberry
x_0 (g kg^{-1})	A*	42.29 ± 0.86^{c}	52.69 ± 2.45^{b}	55.77 ± 3.16^{b}	54.20 ± 2.63^{b}	65.74 ± 1.32^{a}
	D	47.82 ± 0.96^{ab}	44.98 ± 1.52^{bc}	42.58 ± 0.03^{c}	47.47 ± 1.04^{ab}	51.66 ± 0.02^{a}
k	A	0.886 ± 0.002^{c}	0.914 ± 0.002^{ab}	0.935 ± 0.007^{a}	0.920 ± 0.007^{ab}	0.909 ± 0.002^{b}
	D	0.860 ± 0.003^{c}	0.929 ± 0.004^{b}	0.968 ± 0.001^{a}	0.935 ± 0.002^{b}	0.934 ± 0.000^{b}
C	A*	03.75 ± 0.01^{a}	01.62 ± 0.34^{b}	01.49 ± 0.19^{b}	01.90 ± 0.04^{b}	01.28 ± 0.04^{b}
	D	16.03 ± 0.62^{ab}	27.63 ± 7.45^{a}	05.51 ± 0.55^{b}	11.04 ± 2.42^{b}	08.76 ± 0.21^{b}
R^2	A	0.997	0.999	0.999	0.999	0.999
	D	0.997	0.992	0.999	0.997	0.997

Model ranges are 10≤RH (%)≤90, A – Adsorption, D – Desorption, x_0 – monolayer moisture load, C – monolayer energy constant, k –multilayer energy constant.
* Adsorption isotherms differ significantly from Desorption isotherms (P<0.05). Mean values (± deviation, n=2) in a row with different superscripts differ significantly (P<0.05).

Figure captions

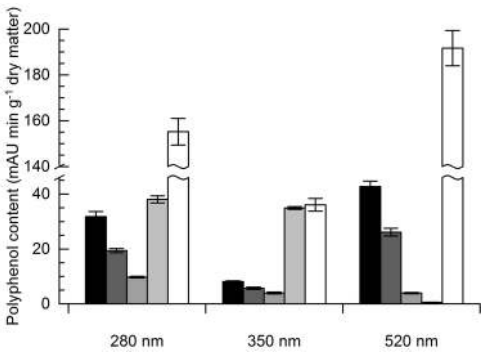
Figure 1. Polyphenol content of freeze-dried berry pomace, representing total polyphenols at 280 nm, flavonols at 350 nm, anthocyanins at 520 nm (*n*=3). ■ *Ribes nigrum* (blackcurrant), ■ *Ribes rubrum* (redcurrant), ■ *Ribes uva-crispa* (gooseberry), ■ *Sorbus aucuparia* (rowanberry), □ *Aronia melanocarpa* (chokeberry)

Figure 2. CIE-Lab colour coordinates for berry pomace powder. BC - *Ribes nigrum* (black currant), RC – *Ribes rubrum* (red currant), GB – *Ribes uva-crispa* (gooseberry), RB – *Sorbus aucuparia* (rowanberry), CB – *Aronia melanocarpa* (chokeberry).

Figure 3. FESEM images of dried and milled pomace particles. BC - *Ribes nigrum* (black currant), RC – *Ribes rubrum* (red currant), GB – *Ribes uva-crispa* (gooseberry), RB – *Sorbus aucuparia* (rowanberry), CB – *Aronia melanocarpa* (chokeberry).

Figure 4. Light microscopy images of dried and milled pomace particles. BC – *Ribes nigrum* (black currant), CB – *Aronia melanocarpa* (chokeberry). Left column: bright field, right column: fluorescence.

Figure 5. Water vapour sorption isotherms of berry pomace powder at 20 °C left: ■ *Ribes nigrum* (blackcurrant), ■ *Ribes rubrum* (redcurrant), ■ *Ribes uva-crispa* (gooseberry); right: ■ *Sorbus aucuparia* (rowanberry), ■ *Aronia melanocarpa* (chokeberry); Adsorption – full line, Desorption – dotted line; error bars represent deviation (n=2)



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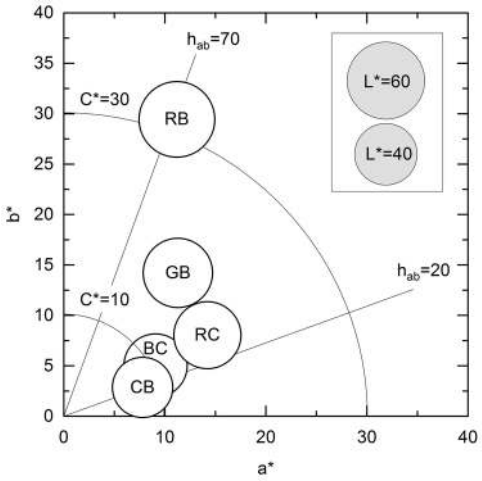

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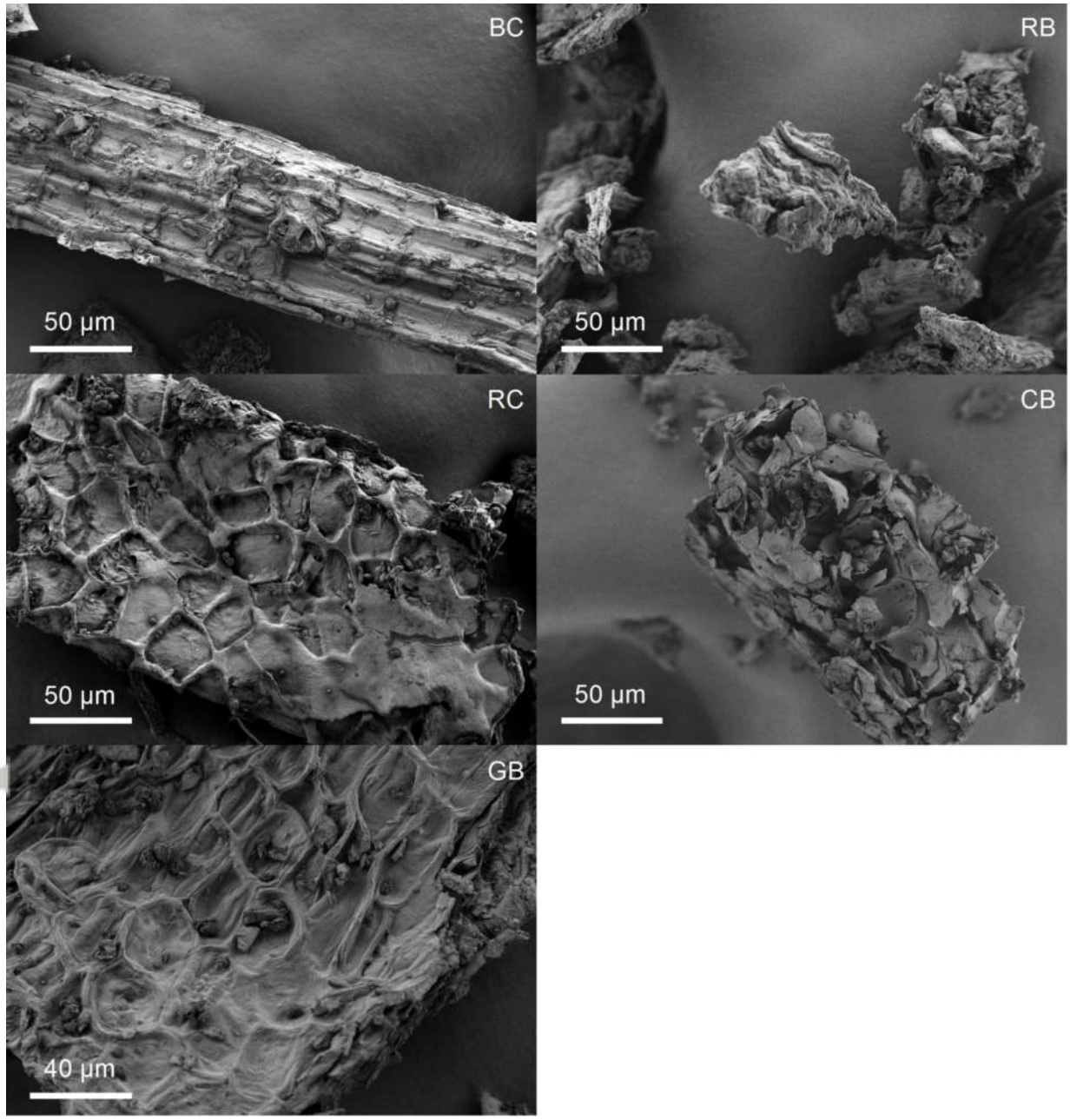

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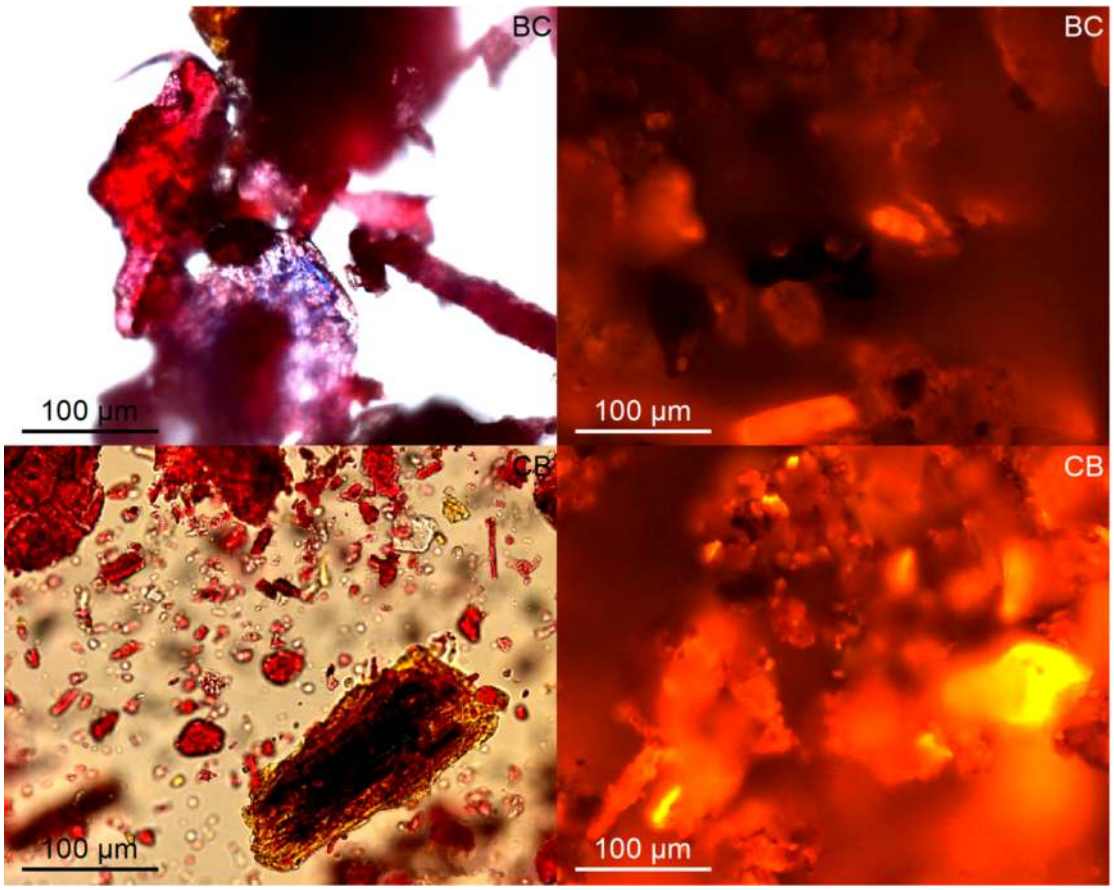

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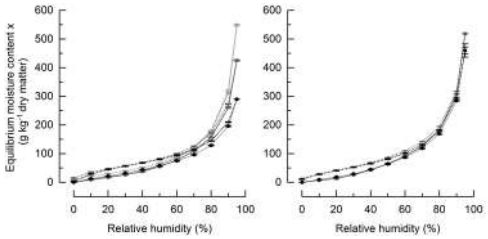

Figure5.TIF