

# Effect of dehydration on phenolic compounds and antioxidant activity of blackcurrant (Ribes nigrum L.) pomace

Article

Accepted Version

Azman, E. M., House, A., Charalampopoulos, D. ORCID: https://orcid.org/0000-0003-1269-8402 and Chatzifragkou, A. ORCID: https://orcid.org/0000-0002-9255-7871 (2021) Effect of dehydration on phenolic compounds and antioxidant activity of blackcurrant (Ribes nigrum L.) pomace. International Journal of Food Science and Technology, 56 (2). pp. 600-607. ISSN 1365-2621 doi: 10.1111/ijfs.14762 Available at https://centaur.reading.ac.uk/92089/

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To link to this article DOI: http://dx.doi.org/10.1111/ijfs.14762

Publisher: Wiley-Blackwell

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1	Effect of dehydration on phenolic compounds and antioxidant activity
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4	Running title: Effect of drying on blackcurrant phenolics
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#### 20 Abstract

21 This study examined the effect of dehydration on the phenolic compounds and antioxidant 22 activity of blackcurrant (Ribes nigrum L.) pomaces (DBP) subjected to hot air oven 23 drying (HOD), industrial rotary drying (IRD) and freeze drying (FD). Temperature and 24 residence time were evaluated for HOD, whereas air-on and air-off temperature, ratio of 25 drum rotor speed to air speed and particle size were evaluated for IRD. The highest total 26 anthocyanins (ATC) and flavonols (FLV) were obtained in particle size of > 5.0 mm using 27 IRD at 475°C/97°C (air-on/air-off) and higher ratio of drum rotor speed to air speed. 28 Smaller size particles were found susceptible to degradation due to high temperature and 29 retention time applied in IRD, resulting in loss of phenolic compounds in DBP, thus HOD 30 was deemed more suitable. Overall, drying method selection and parameters of operation 31 are key in preserving the concentrations of individual HCA and FLV in DBP.

32 **Keywords:** Blackcurrant pomaces; phenolic compounds; antioxidant activity;

33 rotary drying; particle size distribution

#### 34 INTRODUCTION

Waste management is an essential aspect given the extensive production of plantbased by-products that are often marketed as animal feed (Ajila et al., 2012). Improper management of by-products contributes to high amount of waste and pollutants, which negatively affect the environment (Dubey, 2020). The potential of by-products valorisation as value-added alternatives have recently gained growing attention (O'Shea et al., 2012).

41 In 2017 alone, 11,000 tonnes of blackcurrants were produced in the UK (IBA, 42 2018). Blackcurrant skins, which are typically treated as residues from blackcurrant juice 43 processing, are rich in polyphenols and anthocyanins (ATC) (250 mg/100 g of berries) 44 (Vagiri, 2011) as well as flavonols (FLV) and phenolic acids that are linked to high 45 antioxidant activity (Szajdek & Borowska, 2008). The content of ATC in blackcurrant 46 skins is higher than in blackcurrant flesh and seeds. Both FLV and phenolic acids are 47 particularly valuable as dietary supplements or food additives (Lapornik et al., 2005). The 48 extraction of bioactive components from dried mass is more effective than the extraction 49 of these components from fresh mass (Karam et al., 2016), due to phytochemical 50 degradation process occurring more rapidly in high water activity environment. Moisture content of by-products within the range of 6% to 11% (w/w) (Yang et al., 2013) is 51 52 suggested for higher stability of phytochemicals in pigments, restrained microbial 53 growth, and minimal browning reactions of enzymatic and non-enzymatic origin.

54 Unlike microwave drying methods and combined convective microwave drying 55 methods, the use of convective drying method has been reported to contribute to the linear 56 degradation of ATC, FLV, hydroxycinnamic acids (HCA) (e.g. chlorogenic acids), and 57 antioxidant capacity in blackcurrant pomace (Michalska et al., 2017b). Convective drying 58 can lead to higher amounts of polyphenols and radical scavenging activity than

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conventional drying (Bustos et al. 2018). However, studies on the effect of industrial
rotary drying (IRD) on phenolic compounds and antioxidant activity of blackcurrant
pomaces remain scarce.

This study examined the effect of hot air oven drying (HOD), IRD, and freeze drying (FD) on phenolic compounds, specifically ATC, HCA and FLV, and antioxidant activity of dried blackcurrant (*Ribes nigrum* L.) pomace (DBP). HOD and IRD represented hot air-drying methods, whereas FD served as control. The temperature and residence time were evaluated for HOD, whereas air-on and air-off temperature, ratio of drum rotor speed to air speed, and particle size were evaluated for IRD.

#### 68 MATERIALS AND METHODS

#### 69 Chemicals and solvents

Methanol (99.9%) and hydrochloric acid (HCl, 37%), used in the extraction process, were of analytical grade. Methanol was acquired from Sigma-Aldrich (UK) whereas HCl was acquired from Fisher Scientific (Loughborough, UK). Folin-Ciocalteu reagent, sodium carbonate, and 1,1-diphenyl-2-picryl-hydrazyl (DPPH) were similarly acquired from Sigma-Aldrich (UK). A stock solution of 2 mM DPPH in methanol was prepared.

ATC standards of cyanidin-3-O-glucoside (C3G, 96%), cyanidin-3-O-rutinoside (C3R, 96%), delphinidin-3-O-glucoside (D3G, 95%), delphinidin-3-O-rutinoside (D3R, 95%), kaempferol-3-O-glucoside (K3G, 99%), kaempferol-3-O-rutinoside (K3R, 98%), myricetin-3-O-glucoside (MY3G, 99%), and quercetin-3-O-rutinoside (QU3R, 99%) were acquired from ExtraSynthese Ltd (Genay, France). Caffeic acid (98%), ferulic acid (99%), kaempferol (KA, 99%), myricetin (MYR, 98%), *p*-coumaric acid (98%), quercetin (QU, 95%), and quercetin-3-O-glucoside (QU3G, 98%) were acquired from 83 Sigma-Aldrich (UK). Purified water acquired using a Purite reverse osmosis system
84 (Oxon, UK), was utilised in sample preparation.

#### 85 Sample preparation

A&R House (BCL) Ltd (Bleadon, Weston-super-Mare, UK) kindly supplied both fresh
and dried samples of pressed blackcurrants pomaces from the processing of blackcurrant
juice for the use of this study.

89 *Freeze drying (FD- control).* 40.0 g fresh blackcurrant pomace were lyophilised 90 (Virtis SP Scientific Model 2KBTES, Stone Ridge, New York) at  $-45 \pm 2^{\circ}$ C for 48 h and 91 was used as the control.

Hot air oven drying (HOD). 40 g of fresh blackcurrant pomace were placed on a
tray (23 cm × 33 cm) and dried (SalvisLab Thermocenter TC-40T, Rotkreuz,
Switzerland) at various temperatures (70°C to 120°C) and two separate residence times,
(15 min- short and 30 min-long) and moisture content of samples was recorded with a
Halogen Moisture Analyser (HE73, Mettler Toledo, Greifensee, Switzerland).

*Industrial rotary drying (IRD).* Dried samples of blackcurrant pomaces subjected
to different drying parameters of IRD were received from A&R House (BCL) Ltd
(Bleadon, Weston-super-Mare, UK) (Table S1). Sample A was sieved to different
particle sizes (< 0.8 mm, < 5.0 mm, and > 5.0 mm) and a mixture of all particle sizes at a
ratio of 1:1:1 (w/w/w) (Mix).

For the preparation of DBP samples, the seeds of blackcurrant samples were removed using a coffee blender by grinding for 30 s to pass through a 0.841 mm (20 mesh) sieve (Michalska et al. 2017a). All samples were separated into polyethylene bags and stored at -20°C for further analysis.

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#### 106 Extraction of phenolic compounds

107 ATC, HCA and FLV were extracted based on Bao et al. (2005) with sling modifications, 108 in a sample to solvent ratio of 1:10 (w/v). 2.0 g of ground DBP were added into 20 mL 109 of 1% (v/v) HCl in methanol before the mixture was shaken at 180 rpm and 30°C for 24 110 h. The coloured liquid was vacuum filtered through Whatman No. 1 filter paper 111 (Whatman, Buckinghamshire, UK) using a Buchner funnel to separate the supernatants and residues and 20 mL of fresh solvent was added to the solids for another 24 h. 112 113 Supernatants were pooled together and kept at -20 °C for further analysis. The flow 114 diagram of different drying methods followed by extraction procedures of blackcurrant 115 pomaces is shown in Figure S1.

#### 116 Identification of phenolic compounds by liquid chromatography-mass

#### 117 spectrometry (LC-MS)

118 The ATC, HCA and FLV profile of freeze dried DBP was obtained (Table S2) 119 using a Thermo Scientific Accela HPLC system with a photo diode array (PDA) detector 120 interfaced to a Thermo Scientific LTQ Orbitrap XL mass spectrometer and electrospray 121 ionisation (ESI). Chromatography was carried out on a Zorbax C18 column ( $250 \times 4.6$ 122 mm i.d., particle size 5 µm, Agilent) at 25°C. A binary mobile phase was composed of 123 eluent A (acetonitrile/water/formic acid; 5: 92: 3; v/v/v) and eluent B (0.1% formic acid 124 in acetonitrile), in a flowrate of 1.0 mL/min, with 10 10 µL injection volume, on the following gradient elution: 0-20 min, 5 to 25% B; 20-26 min, 25 to 35% B; 26-28.5 125 126 min, 35 to 55% B; 28.5-32 min, 55 to 95% B; 32-42 min, 95 to 5% B. The detections 127 were carried out at 520 nm (ATC), 360 nm (FLV) and 320 nm (HCA). Approximately 128 75% of the analysed sample was diverted to waste using a post PDA splitter. Another 129 25% was directed into the MS which was operated using an Orbitrap detector in positive 130 and negative ion modes scanning from m/z 85 to m/z 2000, at a scan resolution of 131 100,000. To obtain the conformation data, the samples were also directly infused into the
132 same MS using similar acquisition settings, and the ions of interest were subjected to
133 MS<sup>2</sup>. The MS analysis Qual Browser of Xcalibur software (Thermo Scientific, USA) was
134 used to analyse the acquired data.

#### 135 Quantification of phenolic compounds by high performance liquid

#### 136 chromatography (HPLC)

137 A Zorbax C18 column (250 mm × 4.6 mm i.d., particle size of 5 µm, Agilent) in an 1260 138 Infinity HPLC system (Agilent Technologies, Waldbronn, Germany), equipped with a 139 diode-array detector (DAD), was used to measure the concentration of phenolic 140 compounds in the extracts at 30°C. The mobile phase contained 5% (v/v) formic acid in 141 Milli-Q system (Millipore, Billerica, MA, USA) (solvent A) and 100% (v/v) methanol 142 (solvent B) whereas the gradient elution system involved 15% (B) at 0 min and followed 143 by 35% (B) at 15 min, 60% (B) at 30 min, and finally, 80% (B) at 40 min. The flow rate 144 was set at 1.0 mL/min, while injection volume was fixed at 20  $\mu$ L. The period of analysis 145 was 50 min. The ATC, FLV and HCA were concurrently detected at varying wavelengths 146 (520, 360, and 320 nm, respectively) (Figure S2). The quantification of individual 147 phenolic compounds was carried out using external standard calibration curves.

#### 148 Total phenols

Folin-Ciocalteu method (Waterman & Mole, 1994) was slightly modified for this study to determine total phenols. 20  $\mu$ L of appropriately diluted extracts, 1.58 mL of distilled water, and 100  $\mu$ L of Folin–Ciocalteu reagent were mixed and left for 8 min before 300  $\mu$ L of sodium carbonate (75 g/L) was added. After 2 h of incubation at 25°C, the absorbance of the samples was measured at 765 nm against a blank sample (water sample) to obtain the average values in terms of milligram of gallic acid equivalent per 1 g of dried weight (mg GAE/g DW). Gallic acid (0–100 mg/L) served as the standard ofcalibration curve.

#### 157 **Total antioxidant activity**

Total antioxidant activity of DBP extracts was measured according to Blois (1958), with slight modifications. 200  $\mu$ L of 50-fold diluted extracts and 2 mL of 2 mM methanolic solution of DPPH were mixed and kept in the dark at 30°C for 30 min before the absorbance of the samples was measured at 517 nm. The inhibition (in percentage) was determined based on the following equation:

163 Inhibition (%) = 
$$\frac{A_{\rm o} - A_{\rm e}}{A_{\rm o}} \times 100$$

164

165 where *Ao* denotes absorbance of the control and *Ae* denotes absorbance of the sample.

#### 166 Statistical analysis

Minitab V.16 (Minitab Inc., State College, Pennsylvania, USA) was used for data analysis. Apart from one-way analysis of variance (ANOVA), Tukey's multiple range tests were also performed at 0.05 level. In addition, Pearson correlation was conducted to examine the correlations of phenolic compounds (total ATC, FLV and HCA), total phenols, and antioxidant activity of DBP.

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#### 173 **RESULTS AND DISCUSSION**

#### 174 Hot air oven drying (HOD)

175 The initial moisture content of fresh blackcurrant pomaces was 59.82% (w/w). Moisture

176 contents in DBP were varied from 0.78% to 32.65% (w/w) after HOD (Figure S3).

177 However, only DBP samples with moisture content less than 10% (w/w) were considered

178 for further analysis, as suitable to avoid microbial contamination and quality deterioration 179 (Michalska et al. 2017a). ATC was the main phenolic compound ( $\approx 66\%$ , R = 0.660) found 180 in DBP. D3R was found to be highest, followed by C3R, D3G, and C3G (p<0.05) (Figure 181 **1a**). When the temperature exceeded 100°C for 30 min, the ATC content significantly 182 declined (1.0–1.3-fold) (p < 0.05) due to their thermal sensitivity (Patras et al., 2010). 183 Moreover, higher drying temperature leads to ATC degradation in shorter time (Bustos 184 et al. 2018). Likewise, Sadilova et al. (2006) revealed no correlation between moisture 185 content and total ATC. In other words, temperature and residence time during HOD 186 substantially affect the yield of ATC, regardless of the sample's moisture content. On the 187 other hand, relatively higher ATC content in freeze-dried DBP suggests that minor 188 modifications during lyophilisation process can prevent the degradation of thermally 189 sensitive pigments such as ATC (Sablani et al., 2011).

190 DBP samples dried at 110°C for 15 min had moisture content of 8.23% (w/w) and 191 a relatively higher total HCA (Figure 1b). p-Coumaric acid was dominant (p < 0.05), 192 followed by caffeic and ferulic acid. Between these three compounds, p-coumaric and 193 caffeic acid were found higher at 110°C-15 min, whereas ferulic acid was highest in FD 194 sample. Ferulic acid is heat-sensitive and suspectable to oxidation during conventional 195 heating methods (Li et al., 2009). In the current study, dehydration of blackcurrant 196 pomaces in FD prevented the deterioration of ferulic acid. The moisture content and total 197 HCA were found to be moderately correlated (R = 0.560, p < 0.05). This implies that 198 drying at  $> 100^{\circ}$ C overheated DBP which resulted in HCA deterioration. This is in line 199 with Bustos et al. (2018), who stated that drying berries at 50°C-48 h, 65°C-20 h and 200 130°C–2 h reduced the moisture contents to same levels, but temperatures of 50°C and 201 130°C resulted in the degradation of phenolic compounds due to long processing time 202 and high drying temperature, respectively.

203 DBP samples dried in HOD between 80°C and 120°C for 15 min and 30 min had higher FVL content compared to FD. FLV content was the highest in DBP samples dried 204 205 at 110°C for 15 min (Figure 1c). No correlation could be made between total moisture 206 and FLV content, confirming that the drying parameters in HOD affect the content of 207 FLV in DBP, regardless of the moisture content. This study further identified that in 208 HOD, the concentration of individual FLV was as follows: MY3G > QU3R > QU3G. 209 However, no significant difference was recorded between these FLV in freeze-dried DBP. 210 Zhang et al. (2019) reported that MY3G increased and QU3R decreased during drying 211 pre-treatment at 75°C in Dryopteris erythrosora leaves, whereas QU3R has better heat 212 stability than QU3G (Rohn et al. 2007).

213 The drying process affects total ATC and total phenols as well as the antioxidant 214 activity of the processed sample. As shown in Figure 2a, both total phenols and 215 antioxidant activity were found similar in all drying conditions despite the low content of 216 ATC, HCA and FLV in freeze-dried DBP samples. FD, unlike hot air-drying methods, 217 preserves better certain thermally sensitive phenolic compounds (Sogi et al. 2013). 218 Although HOD offers homogenous drying temperature, the drying process may overheat 219 smaller particles at a certain point. Furthermore, Spigno et al. (2007) found that FD did 220 not degrade total phenolic compounds and reduce the antioxidant activity in grape marc. 221 Besides that, Argyropoulos et al. (2011) reported lower shrinkage (from 5% to 15%) and 222 insignificant collapse (lower than 10%) for berries during FD.

HOD appeared to degrade ATC or sugar moieties into smaller molecules, such as aldehydes, and monomeric phenolic acids or their corresponding anthocyanidins, respectively (Keppler & Humpf, 2005; Fleschhut et al., 2006). Michalska et al. (2017b) reported an exponential formation of hydroxymethylfurfural (HMF) in blackcurrant pomace after drying at > 80°C. Also, overheating of DBP samples potentially extracts

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phenolic compounds and reducing sugars, proteins, and organic acids that can react with the Folin-Ciocalteu reagent, as shown by medium correlations between total ATC and total phenols (R = 0.660, p < 0.05) and total phenols and antioxidant activity (R = 0.784, p < 0.05). Nevertheless, total ATC and antioxidant activity were not correlated, implying that HMF, reducing sugars, proteins, and organic acids contribute more to the antioxidant activity, rather than total ATC alone.

#### 234 Industrial rotary drying (IRD)

235 Fresh blackcurrant pomaces were subjected to different drying parameters in IRD and hot 236 air was rapidly raised from 25°C to 450°C or 475°C (air-on temperature) and around 97°C 237 (air-off temperature) at the end of the drying process, where blackcurrant pomaces were 238 adequately dried. The moisture content of the samples ranged from 7.62% to 8.77% 239 (w/w). The results in Figure 3a demonstrated that the increase in the ratio of drum rotor 240 speed to air speed significantly increased ATC content (p < 0.05). However, the slight 241 differences in the air-on and air-off temperature did not exhibit any changes on ATC 242 content. On the other hand, freeze-dried DBP recorded the lowest ATC content. In IRD, 243 the residence time of particles can be reduced due to the increase in the ratio of drum 244 rotor to air speed. With that, overheating of blackcurrant pomaces and, hence, loss of 245 ATC can be prevented. HCA degraded during gradient heating but increased significantly 246 (p < 0.05) in freeze-dried DBP (Figure 3b). Moreover, the content of *p*-coumaric acid 247 was found high in DBP samples that were dried in IRD (p < 0.05), followed by caffeic 248 and ferulic acid. (Figure 3c), The hot air in IRD appeared to assist the extraction of FLV, 249 as compared to FD. MYR was the predominant FLV compound found in DBP, followed 250 by QU. Additionally, the concentration of MY3G was significantly higher (p > 0.05) than 251 QU3G and QU3R in IRD. The decrease in the ratio of drum rotor speed to air speed 252 caused longer residence time during blackcurrant pomace processing. Consequently,

lower concentrations of QU3G than QU3R were detected due to degradation of quercetin glycosides to their corresponding aglycones, but QU3R was more stable against heat treatment. Rohn et al. (2007) also revealed that roasting temperature, period, and sugar moiety attached to the flavonol aglycone affected the degradation kinetics of onion quercetin glucosides. However, there was no significant differences between MY3G, QU3G and QU3R in the freeze-dried DBP.

259 Total ATC was not correlated with the total phenols and antioxidant activity, 260 despite the high content of total ATC in DBP samples that were dried in IRD under 261 varying conditions (Figure 2b). Nevertheless, the results of Pearson correlation revealed 262 that total HCA and total phenols in DBP samples were strongly correlated (R = 0.840, p 263 < 0.05). Meanwhile, the antioxidant activity was found to be strongly correlated with the 264 total HCA (R = 0.791, p < 0.05) and total phenols (R = 0.875, p < 0.05). The results 265 clearly demonstrated that the high antioxidant activity is most likely linked to the total 266 HCA in freeze-dried DBP samples.

#### 267 Different particle sizes of DBP from industrial rotary drying (IRD)

268 The following drying conditions in the IRD were applied for the DBP samples: (1) air-on 269 temperature of 450°C; (2) air-off temperature of 97°C; (3) decrease in the ratio of drum 270 rotor speed to air speed. The dried DBP samples were then separated into different 271 particle sizes. The moisture content of the samples for each category of particle size was 272 measured: (1) moisture content of 8.34% (w/w) for particle size of > 5.0 mm; (2) moisture 273 content of 8.83% (w/w) for particle size of < 5.0 mm; (3) moisture content of 6.96% 274 (w/w) for particle size of < 0.8 mm; (4) moisture content of 8.06% (w/w) for the mixtures 275 of all particle sizes (Mix). The DBP particle size was presumed to have an effect on ATC 276 and other phenolics content.

The content of ATC in DBP was found significantly (p < 0.05) higher for particle size > 5.0 and was the lowest for particle size < 0.8 mm (**Figure 4a**). Lower ratio of drum rotor speed to air speed seems to allow particles of larger size to undergo efficient mass and heat transfer, while particles of smaller size may experience overheating. In this case, the residual temperature of smaller particles potentially exceeds the air-off temperature.

282 Total HCA in DBP appeared to be the highest (p < 0.05) for all particle sizes 283 (Figure 4b). *p*-Coumaric acid was the dominant HCA component in DBP (p < 0.05), 284 regardless of the particle size. On the other hand, the results for total ATC (Figure 4a) 285 and total FLV (Figure 4c) in DBP samples for varying particle sizes were similar. 286 Although the particles with size of > 5.0 mm and < 5.0 mm tolerated higher residence 287 time (lower ratio of drum rotor speed to air speed), higher moisture content was recorded, 288 which was reaffirmed by the significant correlation between moisture content and total 289 ATC (R = 0.645, p < 0.05) and FLV (R = 0.818, p < 0.05) in DBP. Higher moisture content suggests non-overheated particles and successful preservation of thermally 290 291 sensitive phenolic compounds, such as ATC and FLV. Samples with particle size of > 5.0292 mm showed higher concentration of MY3G than QU, while particle sizes of < 0.8 mm, <293 5.0 mm and the mix had higher concentrations of QU than MY3G. Overheating of smaller 294 particle sizes might lead to rapid degradation of QU3G and QU3R and QU aglycone 295 production (Deng et al. 2011). DBP samples exhibited varying amounts of total phenols 296 for different particle sizes, which can be explained by the exposure of surface area to the 297 hot air in the rotary dryer (Figure 2c). Despite the above results, particle sizes of > 5.0298 mm and < 5.0 mm appeared to be poorly correlated with total ATC, FLV and HCA as 299 well as total phenols. However, the correlation between total phenols and antioxidant 300 activity was intermediate (R = 0.691), which may be due the high drying temperature and 301 shorter drying time for DBP. Such drying conditions potentially reduce substances and nitrogen-containing compounds that can react with the Folin-Ciocalteu reagent, resulting
in higher antioxidant activity (Escarpa & González, 2001; Michalska et al., 2016; Bustos
et al., 2018).

#### 305 CONCLUSIONS

306 This study successfully demonstrated that the application of HOD at lower temperature 307 and longer residence time prevents the degradation of total ATC, whereas higher 308 temperature and shorter residence time (110°C–15 min) prevents the degradation of total 309 HCA and FLV in DBP. Meanwhile, the increase in air-on (475°C) and air-off temperature 310 (97°C) and the ratio of drum rotor speed to air speed were found to directly increase the 311 contents of ATC and FLV. However, the application of IRD was found not appropriate 312 for thermally sensitive HCA. Particles of smaller size are more likely to be damaged by 313 high temperature and retention time in IRD, resulting in the loss of phenolic compounds. 314 The application of FD efficiently retains thermally sensitive phenolic compounds and 315 non-phenolic compounds with high antioxidant activity such as HCA.

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#### 317 ACKNOWLEDGEMENTS

The authors are gratefully acknowledged the Malaysian Higher Education and Universiti Putra Malaysia (UPM) for the financial support in this research. Also, we acknowledge A & R House (BCL) Ltd, (Bleadon, Weston-super-Mare, UK) for the kind supply of blackcurrants by-products.

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#### 323 ETHICS APRROVAL STATEMENT

324 Ethics approval was not required for this research

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#### 326 CONFLICT OF INTEREST

327 The authors have declared no conflicts of interest for this article.

328

#### 329 DATA AVAILABILITY STATEMENT

330 Data available on request from the authors

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