

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

Article

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

3
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

35 Waste management is an essential aspect given the extensive production of plant-
36 based by-products that are often marketed as animal feed (Ajila et al., 2012). Improper
37 management of by-products contributes to high amount of waste and pollutants, which
38 negatively affect the environment (Dubey, 2020). The potential of by-products
39 valorisation as value-added alternatives have recently gained growing attention (O'Shea
40 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
51 content of by-products within the range of 6% to 11% (w/w) (Yang et al., 2013) is
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

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

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

68 **MATERIALS AND METHODS**

69 **Chemicals and solvents**

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

76 ATC standards of cyanidin-3-O-glucoside (C3G, 96%), cyanidin-3-O-rutinoside
77 (C3R, 96%), delphinidin-3-O-glucoside (D3G, 95%), delphinidin-3-O-rutinoside (D3R,
78 95%), kaempferol-3-O-glucoside (K3G, 99%), kaempferol-3-O-rutinoside (K3R, 98%),
79 myricetin-3-O-glucoside (MY3G, 99%), and quercetin-3-O-rutinoside (QU3R, 99%)
80 were acquired from ExtraSynthese Ltd (Genay, France). Caffeic acid (98%), ferulic acid
81 (99%), kaempferol (KA, 99%), myricetin (MYR, 98%), *p*-coumaric acid (98%),
82 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**

86 A&R House (BCL) Ltd (Bleadon, Weston-super-Mare, UK) kindly supplied both fresh
87 and dried samples of pressed blackcurrants pomaces from the processing of blackcurrant
88 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 $-45 \pm 2^{\circ}\text{C}$ for 48 h and
91 was used as the control.

92 *Hot air oven drying (HOD)*. 40 g of fresh blackcurrant pomace were placed on a
93 tray (23 cm \times 33 cm) and dried (SalvisLab Thermocenter TC-40T, Rotkreuz,
94 Switzerland) at various temperatures (70°C to 120°C) and two separate residence times,
95 (15 min- short and 30 min-long) and moisture content of samples was recorded with a
96 Halogen Moisture Analyser (HE73, Mettler Toledo, Greifensee, Switzerland).

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

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

106 **Extraction of phenolic compounds**

107 ATC, HCA and FLV were extracted based on Bao et al. (2005) with slight 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
112 and residues and 20 mL of fresh solvent was added to the solids for another 24 h.
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 × 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 µL injection volume, on the
125 following gradient elution: 0–20 min, 5 to 25% B; 20–26 min, 25 to 35% B; 26–28.5
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 MS². 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 μL. 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**

149 Folin-Ciocalteu method (Waterman & Mole, 1994) was slightly modified for this study
150 to determine total phenols. 20 μL of appropriately diluted extracts, 1.58 mL of distilled
151 water, and 100 μL of Folin–Ciocalteu reagent were mixed and left for 8 min before 300
152 μL of sodium carbonate (75 g/L) was added. After 2 h of incubation at 25°C, the
153 absorbance of the samples was measured at 765 nm against a blank sample (water sample)
154 to obtain the average values in terms of milligram of gallic acid equivalent per 1 g of

155 dried weight (mg GAE/g DW). Gallic acid (0–100 mg/L) served as the standard of
156 calibration curve.

157 **Total antioxidant activity**

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

$$163 \quad \text{Inhibition (\%)} = \frac{A_o - A_e}{A_o} \times 100$$

164

165 where A_o denotes absorbance of the control and A_e denotes absorbance of the sample.

166 **Statistical analysis**

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

172

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 susceptible 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 $> 100^\circ\text{C}$ 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
204 higher FVL content compared to FD. FLV content was the highest in DBP samples dried
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.

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

228 phenolic compounds and reducing sugars, proteins, and organic acids that can react with
229 the Folin-Ciocalteu reagent, as shown by medium correlations between total ATC and
230 total phenols ($R = 0.660$, $p < 0.05$) and total phenols and antioxidant activity ($R = 0.784$,
231 $p < 0.05$). Nevertheless, total ATC and antioxidant activity were not correlated, implying
232 that HMF, reducing sugars, proteins, and organic acids contribute more to the antioxidant
233 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,

253 lower concentrations of QU3G than QU3R were detected due to degradation of quercetin
254 glycosides to their corresponding aglycones, but QU3R was more stable against heat
255 treatment. Rohn et al. (2007) also revealed that roasting temperature, period, and sugar
256 moiety attached to the flavonol aglycone affected the degradation kinetics of onion
257 quercetin glucosides. However, there was no significant differences between MY3G,
258 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.

277 The content of ATC in DBP was found significantly ($p < 0.05$) higher for particle
278 size > 5.0 and was the lowest for particle size < 0.8 mm (**Figure 4a**). Lower ratio of drum
279 rotor speed to air speed seems to allow particles of larger size to undergo efficient mass
280 and heat transfer, while particles of smaller size may experience overheating. In this case,
281 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
290 content suggests non-overheated particles and successful preservation of thermally
291 sensitive phenolic compounds, such as ATC and FLV. Samples with particle size of > 5.0
292 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 > 5.0
298 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

302 nitrogen-containing compounds that can react with the Folin-Ciocalteu reagent, resulting
303 in higher antioxidant activity (Escarpa & González, 2001; Michalska et al., 2016; Bustos
304 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.

316

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322

323 **ETHICS APPROVAL STATEMENT**

324 Ethics approval was not required for this research

325

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

331

332

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