

1 An analytical approach to determine the health benefits and health risks of 2 consuming berry juices

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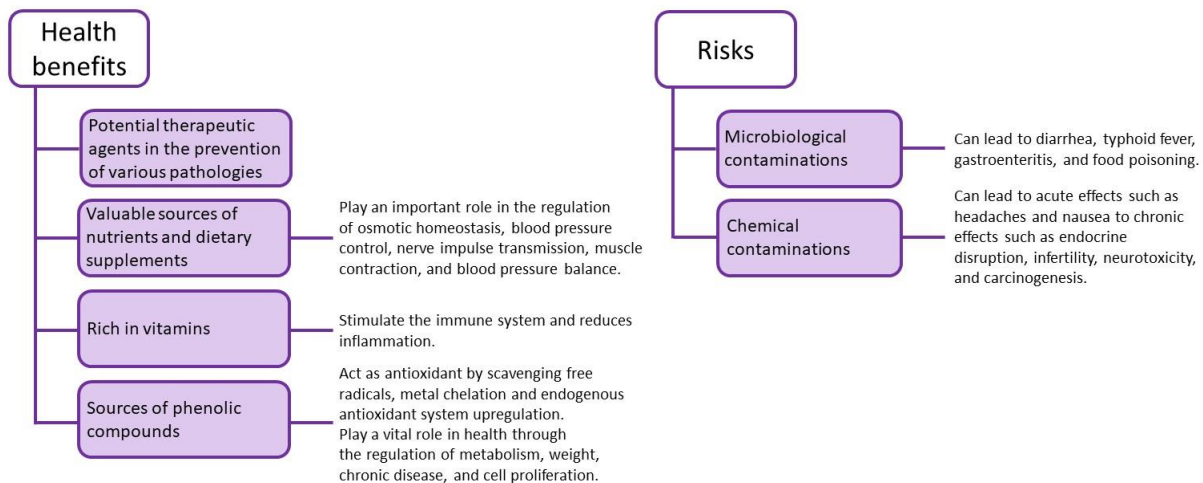
18 ABSTRACT

19 Food products composition analysis is a prerequisite for verification of product quality,
20 fulfillment of regulatory enforcements, checking compliance with national and international
21 food standards, contracting specifications, and nutrient labeling requirements and providing
22 quality assurance for use of the product for the supplementation of other foods. These aspects
23 also apply to the berry fruit and berry juice. It also must be noted that even though fruit juices
24 are generally considered healthy, there are many risks associated with mishandling both fruits
25 and juices themselves. The review gathers information related with the health benefits and risk
26 associated with the consumption of berry fruit juices. Moreover, the focus was paid to the
27 quality assurance of berry fruit juice. Thus, the analytical methods used for determination of
28 compounds influencing the sensory and nutritional characteristics of fruit juice as well as
29 potential contaminants or adulterations.

30 **Keywords:** berry juice; antioxidants; adulteration; health benefits; health risks; green
31 analytical chemistry.

32 1. Introduction

33 Human nutrition science has developed in the last decades, turning from looking at foods
 34 as a simple source of energy to the appreciation of their role in maintaining health and in
 35 reducing disease risks. Nowadays, berry plants have become very attractive to the food industry,
 36 as it is a trend to prompt their application as replacements for synthetic nutraceuticals. In
 37 addition, the berry itself is often used to produce juice (Li et al., 2017). Due to the high content
 38 of polyphenols, antioxidants, and other bioactive compounds, berry fruit juices are often seen
 39 as one of the healthiest and the most nutritious beverages (Skrovankova et al., 2015). The
 40 mentioned groups of compounds are responsible for various health benefits, including
 41 cardiovascular diseases, prevention of inflammation disorders, or protective effects to lower the
 42 risk of various cancers (Figure 1) (de Souza et al., 2014). Because of that general assumption,
 43 it is important to evaluate whether the amount of numerous bioactive ingredients is sufficient
 44 for them to be beneficial to human health. Moreover, it is important to assess the content of
 45 substances such as polyphenols, antioxidants, and vitamins in different juices, since their levels
 46 may differ depending not only on the type of fruit but also on its origin, processing, or storage
 47 (Arfaoui, 2021).



48
 49 Figure 1. Health benefits and risks related to the consumption of berry fruit juices.

50 Even though berry fruit juices are generally considered healthy, there are many risks associated
 51 with mishandling both fruits and juices themselves (Abisso et al., 2018). It is important to
 52 collect information on the negative effects that microbial contamination, inappropriate storing
 53 condition, or the use of stale fruits may have on juices composition and safety of their
 54 consumption. While some of the risks are minimized in the case of commercially available
 55 berry juices, quality assessment is of particular importance in the case of homemade juices,
 56 since consumers are not bound to obey the same sanitary standards as food producers (Li et al.,
 57 2017).

58 One of the most overlooked problems associated with fruit juices, including berry fruit juices
 59 consumption is the possibility of their adulteration (Dasenaki & Thomaidis, 2019). To decrease
 60 the cost of juices production, the producer may mix in fruits that are not only cheaper but also

61 might not have as many health benefits, as well as resorting to the addition of water. Moreover,
62 since juices that are the richest in bioactive compounds are not always seen as the most
63 palatable, additives are used to improve their sensory qualities leading to a further decrease of
64 wholesomeness. Another important adulteration is the application of artificial flavors to mimic
65 the natural aroma (Hrubá et al., 2021).

66 This work aims to assess the current status of analytical approaches to fruit juices analysis and
67 quality assessment to identify both the current limitations and future trends. In the article, we
68 critically review the up-to-date literature on fruit juices analysis, in particular on their quality
69 assessment. The information is provided in a concise and approachable form, focusing on
70 practical aspects and examples of implementation rather than on detailed technical aspects and
71 principles of operation of the described methods. The focus is mainly placed on novel methods
72 for fruit juices quality assessment in the context of green analytical chemistry, but diverse and
73 interesting examples of studies that showcase the possibilities of future developments are
74 presented. In addition, the practicality of the reviewed methods for end-users in and outside of
75 analytical laboratories are given. Current trends and lines of future research are also discussed.

76 **2. Health benefits related to consumption of berry juice fruit**

77 The consumption of berry juice has been widely associated with a decreased risk of certain
78 chronic diseases (Giampieri et al., 2015; Habanova et al., 2019). Several studies report that
79 berry juice have different biological activities *in vitro* and *in vivo* systems, which are related to
80 their bioactive composition (Bakuradze et al., 2019; Giampieri et al., 2015; Toaldo et al., 2015).
81 These juices are excellent sources of vitamins, minerals, and phenolic compounds, especially
82 phenolic acids and anthocyanins (Geraldi et al., 2021). The chemical composition of fruit juices
83 depends especially on the fruit species, maturation, climate, and the treatments to which the
84 fruit and the juices themselves are submitted. In addition, during cultivation, climatic conditions
85 have a direct influence on the chemical quality and the polyphenolic complexity of the fruits
86 (Coelho et al., 2021). The technology employed in juice production can provide different levels
87 of extraction of bioactive compounds. The crushing step contributes to the extraction of
88 phenolic compounds present in berries. It is interesting to note that during the fruit processing,
89 some steps, such as freezing and thawing, can affect the extraction of some valuable
90 components in the grinding and pressing steps, changing the phytochemical composition of the
91 fruit juices (Weber & Larsen, 2017).

92 In addition, other processing steps affect the bioactive composition of juices, such as enzymatic
93 treatment, filtration, clarification, and pasteurization. In enzymatic treatment, pectinolytic
94 enzymes are used to increase productivity of juice. The use of the pectinase enzyme causes
95 pectin degradation, which results in reduced juice viscosity and changes in physicochemical
96 properties, such as total soluble solids, pH, and turbidity (Marsol-Vall et al., 2019). Filtering or
97 clarification is carried out before or after pasteurization. The heat treatment (pasteurization) to
98 which the fruit juices are subjected can also cause reductions in the content of vitamins and
99 polyphenols (Stübler et al., 2020), however, pasteurization is still conventionally used as a
100 procedure to preserve juices from microbial contamination (Marsol-Vall et al., 2019). Martino
101 et al. (2013) report that polyphenol retention and antioxidant activity were significantly higher

102 in grape juice clarified after thermal processing (pasteurization) compared to grape juice
103 clarified before pasteurization. In the same way as pasteurization due to the use of high
104 processing temperatures, the evaporation technique used to concentrate fruit juices can reduce
105 the nutritional value and bioactive properties of the product (Amran & Jusoh, 2016). In contrast,
106 low temperatures are employed in the membrane separation technique used to concentrate fruit
107 juices preserving the most thermosensitive compounds, especially vitamins and polyphenols
108 (Bhattacharjee et al., 2017). In addition to the reported steps, storage conditions such as
109 exposure to heat and light have an important influence on polyphenol retention. During storage,
110 the content of monomeric anthocyanins decreases leading to the polymerization of
111 anthocyanins into more stable compounds (Marsol-Vall et al., 2019)..

112 Knowledge about the processes mentioned above is of paramount importance for the
113 elucidation of the chemical composition of wild fruit juices and the correct correlation with the
114 health benefits promoted by the regular consumption of these beverages, since the concentration
115 of vitamins, minerals and bioactive compounds directly depends on the processes to which the
116 fruits and juices are submitted. Clinical and pre-clinical studies have shown that berry fruits
117 and their juices can act as potential therapeutic agents in the prevention of various pathologies,
118 such as diabetes, neurodegenerative and cardiovascular diseases, and cancer (Wang et al., 2021;
119 B. Yang and Kortensniemi, 2015). The literature studies showed that berry fruits and their juices,
120 alone or in combination with other functional foods or dietary interventions, can improve
121 glycemic and lipid profiles, blood pressure, and surrogate markers of atherosclerosis (Calvano
122 et al., 2019).

123 Berry fruits contain large amounts of essential and physiologically important macroelements
124 such as P, Mg, K, and Na (Szymczycha-Madeja et al., 2014). As well as some microelements
125 such as Ca and Fe (Toaldo et al., 2015). Both macro and microelements play an important role
126 in the regulation of osmotic homeostasis, blood pressure control, nerve impulse transmission,
127 muscle contraction, and blood pressure balance (Gharibzahedi & Jafari, 2017). The daily
128 consumption of fruit juices can be a part of the recommended daily doses of some nutritionally
129 important elements, such as Co, Cr, Cu, Fe, Mn, Ni, Zn, and Se (Szymczycha-Madeja et al.,
130 2014).

131 The berry fruit juices are also rich in vitamins A, C, and E, and vitamins of the B complex,
132 which are essential for health, as their consumption stimulates the immune system and reduces
133 inflammation (Skrovankova et al., 2015). Since inflammation plays a key role in the
134 development of diabetes, asthma, cardiovascular disease, and cancer, the consumption of
135 appropriate amounts of the above-mentioned vitamins reduces the risk of those diseases
136 (Maleki et al., 2019). Trych et al. (2020) reported that black currant contains approximately
137 160–285 mg/100 g of vitamin C. Zheng et al. (2009) reported vitamin C contents of 60–190
138 mg/100 mL in blackcurrant juices. Sapei et al. (2014) reported that the ascorbic acid content of
139 fresh strawberry juices ranged from 20 to 40 mg/100 mL. The consumption of vitamin C is
140 associated with several health benefits, as it has anti-inflammatory, antibacterial, and
141 neuroprotective action.

142 We also highlight that in addition to the composition of minerals and vitamins, wild fruit juices
143 are excellent sources of phenolic compounds, especially anthocyanins and phenolic acids
144 (Bakuradze et al., 2019). It is known that polyphenols present in fruits and vegetables exert
145 beneficial biological activities to the human body when consumed regularly due to their
146 antioxidant, cardioprotective, anti-inflammatory, and neuroprotective activities. It is known that
147 food matrix in which given compounds are present is important factor determining its release
148 and stability while digested in a human body. To become bioavailable and subsequently
149 bioaccessible polyphenols must be removed from the digested matrix and solubilized in the
150 gastrointestinal fluids. Therefore, it is important to say that phenolic compounds exert their
151 health-related properties when they reach the target tissue of the human body in biologically
152 active concentrations (da Silva Haas et al., 2019). Anthocyanins are the most important
153 flavonoids present in berry fruits and contribute to their high antioxidant capacity. These
154 compounds are responsible for the flavor and the red color of the fruits (Cortez et al., 2017).

155 The small and aromatic berries of blackcurrant (*Ribes nigrum*) are rich in anthocyanins
156 (delphinidin 3-glucoside, delphinidin 3-rutinoside, cyanidin 3-glucoside, and cyanidin 3-
157 rutinoside) (Tian et al., 2023), and its consumption inhibits the activities of the dipeptidyl
158 peptidase-enzymes IV, α -amylase, α -glucosidase, nitric oxide synthase, and cyclooxygenase-2
159 which are biochemical markers of type 2 diabetes and inflammation (Kowalski & Gonzalez de
160 Mejia, 2021). Other health benefits were reported by Cortez and Gonzalez De Mejia (2019),
161 such as improved cardiovascular, nervous, ocular, skeletal, skin, and renal systems (Cortez &
162 Gonzalez de Mejia, 2019).

163 The blueberry juice (*Vaccinium ashei*) also has high concentrations of anthocyanins, such as
164 cyanidin-3-glycoside, peonidin-3-glycoside, malvidin-3-glycoside, malvidin-3-galactoside,
165 and malvidin-3-arabinoside (Wu et al., 2021). Yang and Kortensniemi (2015), reported an
166 inverse association between anthocyanin intake and the incidence of chronic disease. Recent
167 research shows that flavonoids can inhibit regulatory enzymes or transcription factors important
168 for the control of mediators involved in inflammation, in addition to attenuating tissue damage
169 and fibrosis (Maleki et al., 2019).

170 Strawberry juice (*Fragaria X ananassa*, Duch.) is one of the berry juices that has been gaining
171 interest for its positive effect on health due to its polyphenol composition, with emphasis on
172 phenolic acids and anthocyanins. Among the acids, ellagic acid stands out, it is a dimeric
173 condensation product of gallic acid and is found naturally in strawberries, raspberries, and
174 blackberries and has important anticancer, antithrombotic, and anti-inflammatory properties
175 (Muthukumar et al., 2017). In addition, strawberries are rich in anthocyanins (pelargonidin-
176 3-glucoside, cyanidin 3-glycoside, and pelargonidin 3-rutinoside) that promote benefits to
177 human health, as they can regulate gene expression and prevent DNA damage (Giampieri et al.,
178 2015). Preclinical and clinical investigations support the role of anthocyanins in ocular health,
179 these polyphenols have been associated with several benefits pertinent to neurodegeneration.
180 The anthocyanins allow the reduction of induced oxidative stress, decreasing the levels of
181 reactive oxygen species, and malondialdehyde and increasing the levels of superoxide
182 dismutase, catalase, and glutathione peroxidase in the pigment epithelium of the human retina
183 (Huang et al., 2018). According to McNamara et al. (2018) supplementation with blueberry

184 powder generated an improvement in the cognitive function of elderly people with subjective
185 cognitive impairment, supposedly derived from a vaso-modulatory effect.

186 On the other hand, the acute consumption of grape juice (*Vitis labrusca* L.) rich in catechin,
187 epicatechin, *trans*-resveratrol, and anthocyanins (cyanidin 3,5-diglucoside and malvidin 3,5-
188 diglucoside) increases the levels of antioxidants in plasma and erythrocytes in healthy
189 individuals, reducing the lipid peroxidation (Toaldo et al., 2016). Grape juice consumption may
190 render additional benefits for healthy adults who exercise regularly. Grape juice had great
191 potential as an antioxidant source in improving the antioxidant status and cardiometabolic
192 profile of healthy adults. Catechin, isoquercetin, and procyanidin B1 were the major compounds
193 in grape juice from cultivars Isabel, Bordô, and Concord. The plasma antioxidant activity and
194 HDL-cholesterol increased after grape juice intake, and LDL-cholesterol and systolic blood
195 pressure decreased after grape juice consumption (Toscano et al, 2017). Renaud and De
196 Lorgeril proposed in 1992 the French paradox which states that the consumption of polyphenols
197 is associated with a low incidence of heart and coronary disease, despite a high-fat diet. It is
198 noteworthy that the understanding of the “French Paradox” has stimulated the interest of further
199 research to investigate whether polyphenols may offer protective effects beyond the
200 cardiovascular system and whether different botanical sources may also offer beneficial effects
201 on human health (Sun et al., 2002).

202 With regards to the stability of phenolic compounds, they are susceptible to several structural
203 changes during gastrointestinal digestion, and among polyphenols, phenolic acids seem to be
204 the most resistant compounds, being the most relevant to explain the biological activity of foods
205 (Corrêa et al., 2017; Lingua et al., 2019). Chlorogenic and protocatechuic acids are the major
206 phenolic acids in blueberry juice (*Vaccinium ashei*) (Wu et al., 2020) and blackberry juice
207 (*Rubus americanus*) (Wu et al., 2021). It is known that polyphenols act in the prevention of
208 oxidative stress, inhibiting inflammation and improving vascular health (Sinopoli et al., 2019).
209 According to Yang and Kortessniemi (2015), regular consumption of polyphenol-rich fruit
210 juices improves the postprandial glycemic response and the profile of circulating inflammatory
211 markers, in addition to increasing plasma antioxidant capacity and delaying the loss of related
212 cognitive functions the age.

213 Growing evidence suggests that wild fruit consumption has significant potential in preventing
214 and treating most risks associated with a metabolic syndrome like diabetes mellitus (Hameed
215 et al. 2020, Vendrame et al. 2016). This is probably due to the presence of polyphenols with
216 known antioxidant and anti-inflammatory effects, such as phenolic acids and anthocyanins. In
217 offering efficient and secure dietary therapies for diabetes mellitus prevention and control,
218 tailored berries nutrition is compared to an individual pharmaceutical strategy (Hameed et al.,
219 2020). In this context, the use of analytical methods to determine the bioactive composition of
220 wild fruit juices is extremely important for the beverage industry, as this way, it is possible to
221 report their nutritional potential and possible health benefits. We emphasize that to elucidate
222 different perspectives on the nutritional potential of food, it is necessary to consider the effect
223 of digestion, absorption, and metabolization of the bioactive compounds in the human body
224 (Attri et al., 2017; Velderrain-Rodríguez et al., 2014).

225 3. Risks associated with consumption of fruit juice

226 The insightful evaluation of the quality of berry fruit juices is of great importance for consumer
227 safety. Although the consumption of fruit juices generally has several positive health effects, it
228 is notorious to emphasize the possible risks associated with microbiological contaminations,
229 characterized by the presence of pathogenic microorganisms, such as bacteria, viruses, and
230 fungi, and chemical contaminations, which mainly include the presence of pesticides,
231 mycotoxins, illegal additives, metals, bisphenols, and organic pollutants (Mostafidi et al.,
232 2020).

233 Quality inspection of fruit juices is governed by different bodies in their respective countries
234 and regions. The European Fruit Juice Association Code of Practice (AIJN COP) brings
235 together a collection of reference guidelines for fruits and vegetables. The AIJN COP contains
236 parameters that a fruit or vegetable juice needs to meet in the European market, establishing
237 criteria for evaluating juices with respect to quality, authenticity, and identity. In addition to the
238 AIJN COP, other bodies establish drink quality criteria, such as the Polish Association of Juice
239 Producers (KUPS) in Poland and the Food and Drug Administration (FDA) in the United States.
240 It should be noted that the SDS requires the application of Hazard Analysis Critical Control
241 Point (HACCP) principles that aim to ensure the safe and sanitary processing of fruit and
242 vegetable juices.

243 3.1. Microbiological Contaminations

244 3.1.1. Bacteria

245 The contamination of fruit juices by bacteria, these occur in places with inadequate facilities
246 and a lack of hygienic-sanitary standards, mainly due to the lack of care when handling the
247 fruits used during juice preparation (Nawawee et al., 2019). The bacteria *Escherichia coli*,
248 *Salmonella typhi*, *Pseudomonas spp*, *Staphylococcus aureus*, and *Vibrio cholerae* are the most
249 common in fruit juices and the consumption of drinks contaminated by these pathogens can
250 lead to diarrhea, typhoid fever, gastroenteritis, and food poisoning (Sharma et al., 2020).

251 3.1.2. Fungi

252 The presence of fungi in fruit juices is an important factor to be considered since fungi are
253 capable of producing mycotoxins, which are toxic secondary metabolites that pose a potential
254 risk to human health (Fliszár-Nyúl et al., 2020). Recent studies have shown that a wide variety
255 of small berries, such as strawberries, blueberries, mulberries, blackcurrants, and raspberries,
256 due to their soft and fragile skin, are susceptible to small lesions that allow the growth of fungi,
257 especially molds. Once present in the fruit, fungi can resist heat treatments used in the
258 processing of fruit juices and persist in the product. In addition, products stored at room
259 temperature are more prone to the occurrence of fungi (Jackson & Al-Taher, 2008). The main
260 fungi found in fruits belong to the genera *Aspergillus*, *Penicillium*, and *Alternariae* (Guo et al.,
261 2021).

262 3.1.3. Virus

263 The norovirus or hepatitis A virus is one of the most common viruses in fruit derivatives, being
264 recognized as the cause of gastroenteritis and hepatitis in humans. Virus contamination of fruit
265 juices is mainly related to the contamination that occurs during the various stages of raw
266 material (fruits) production, including production, harvesting, processing, and distribution
267 (Takahashi et al., 2018). A significant source of viruses can result from water contaminated
268 with the viruses that are used for irrigation during planting, as it is not practical for all fruit
269 farms to use potable water. Furthermore, bacterial indicators employed for water quality control
270 generally cannot predict viral contamination, giving negative results for indicator tests. In
271 addition to water, viral contamination can occur through the hands of fruit handlers without
272 proper hygiene (Maunula et al., 2013).

273 3.2 Chemical contamination

274 Considering the quality and the safety of fruit juices, there is a growing interest in evaluating
275 potential chemical contaminants such as pesticides, bisphenols or metals, as well as the
276 presence of adulterants. Chemical contaminants present in fruit juices are usually derived from
277 the fruit itself, which during crop production, post-harvest, and additional processes are exposed
278 to contaminants like pesticides, metals, etc. In addition, packaging can be a potential source of
279 chemical contaminants (Mostafidi et al., 2020).

280 3.2.1. Pesticides

281 In recent years agriculture has advanced rapidly and intensified the use of a huge amount of
282 chemical inputs, mainly synthetic pesticides that play an important role in the protection of
283 several cultivars, including pest control and disease prevention (Heidari et al., 2020). Pesticides
284 belong to different chemical groups such as carbamates, neonicotinoids, organochlorines,
285 organophosphorus, phenoxyacids, pyrethroids, strobilurins, triazines, triazoles etc.) and have
286 two different modes of action (contact or systemic). Pesticides with contact action accumulate
287 on/in the plant layer, while pesticides with systemic action penetrate deeper into plant tissues,
288 and were definitely more difficult to reduce during juice production (Jankowska et al., 2018).
289 It was proven that after squeezing of blackcurrants, the pesticide residue levels in the juice were
290 lower by more than 50% compared to raw fruits. Moreover, the contact pesticides remained on
291 the peel and minimally penetrated into the juice (18% compared to the raw fruits) (Jankowska
292 et al., 2018). The beverage industry is the fastest-growing food sector worldwide, especially the
293 production of fruit juices (*Food And Beverages Global Market Report 2022- Product Image*
294 *Food And Beverages Global Market Report 2022*, 2022). On the other side, in the latest sector
295 some problems can be pointed out, especially, the application of enormous doses of pesticides
296 used to increase productivity and product quality. As a result, from the use in agricultural
297 activities, residual pesticides can ultimately be found in the human diet, since if they are not
298 naturally degraded, they can penetrate plant tissues and be found in the fruit pulp and later in
299 the juices. In addition, the use of techniques to concentrate processed juices can promote an
300 increase in the pesticide content in the final product when compared to fruits (Jin et al., 2012).

301 Prolonged exposure to pesticides can generate several negative health effects, from acute effects
302 such as headaches and nausea to chronic effects such as endocrine disruption, infertility,
303 neurotoxicity, and carcinogenesis (de-Assis et al., 2020). Within this context, it is important to

304 emphasize that pesticides are regulated to ensure that pesticide residues in food do not pose a
305 risk to human health. Thus, commercialized pesticides are authorized after an intensive
306 assessment of possible health and environmental risks (Torović et al., 2021). The maximum
307 legally permitted concentrations of pesticide residues in specific foods, including fruit juices,
308 are regulated by the European Union (CE/n°299/2008), but also the United States
309 Environmental Protection Agency sets such limits. Furthermore, efforts to minimize the impact
310 of pesticide residues can be adopted through the implementation of practices such as the rational
311 use of pesticides, exploitation of natural pesticides, promotion of organic agriculture, and
312 adequate application intervals (Mostafidi et al., 2020).

313 3.2.2. Metals

314 Fruit juices are an important source of minerals, which are essential for maintaining health
315 (Caswell, 2009). However, in addition to the presence of elements beneficial to health, juices
316 can contain harmful metals such as Hg, Sn, As, and Cd that can trigger serious problems for
317 human health, even at low concentration levels. The consumption of metals through fruit juices
318 can cause chronic diseases or mutagenesis and carcinogenesis (Bhattacharya et al., 2016). In
319 addition to the risk of these elements to human health, the presence of some metals in excess,
320 like Fe and Cu can reduce the shelf life of foods or possibly decrease the nutritional value of
321 juices, since these metals are responsible for catalyzing oxidative processes, through free
322 radicals oxidative deterioration (Mohamed et al., 2020).

323 The presence of unwanted metals in juices can come from the packaging or from the fruit itself.
324 Juices stored in aluminium containers can be contaminated by the metal through leaching
325 processes. Al (III) is highly toxic to humans due to the potential accumulation in the brain that
326 can trigger Parkinson's and Alzheimer's disease (Hafez et al., 2019). The metals derived from
327 fruits are commonly related to the mineral composition of the planting soil, agricultural
328 practices with abusive use of pesticides or contaminants transported by air or water, in this
329 sense, it is extremely important to regularly monitor the dietary intake of food sources to ensure
330 safe food. In addition to ensuring the nutritional value of food (Anastácio et al., 2018).

331 3.2.3. Biogenic amines

332 Biogenic amines (BAs) are aliphatic or aromatic organic compounds of low molecular weights.
333 They are generated during cellular metabolism in bacteria, plants, and animals due to microbial
334 decarboxylation of the corresponding amino acids. The amount and kind of BAs produced are
335 influenced significantly by the food composition, and factors that allow bacteria to flourish
336 during food processing and storage (Gomez-Gomez et al., 2018). Low quantities of BAs in food
337 are not thought to be dangerous, but they may have toxic effects when eaten in large doses.
338 Their analysis in food samples is of tremendous interest not just because of their potential
339 toxicity, but also because they can be utilized as indications of food freshness or rotting (Saaid
340 et al., 2009). Many works have been published according to the determination of BAs in berry
341 juice samples (Sub-section 4.2.2).

342 3.2.4. Adulteration

343 The adulteration of beverages is commonly related to dilution practices, the addition of artificial
344 flavors to mimic the natural aroma, and the addition of flavor masking to alter specific
345 characteristics, such as reducing or eliminating unpleasant flavors such, as bitterness. In
346 addition, the addition of different chemical mixtures capable of masking themselves so that
347 adulteration is not perceived also constitutes adulteration. As a result of adulterations, a lower
348 nutritional value is expected of beverages (Xu et al., 2019).

349 Some examples found in the literature on fruit juice adulteration include the adulteration of
350 grape juice with the addition of fruit juices of lesser commercial value. A good example is the
351 addition of apple juice to whole grape juice. Apples are rich in pectin, which acts as a gelling
352 and natural thickening agent that prevents the separation of the juice phases. Moreover, the
353 addition of apple juice masks other adulterations, including the addition of water and other
354 additives (Oliveira et al., 2019). Similarly, orange juice (*Citrus sinensis*), consumed worldwide,
355 can suffer adulteration by the addition of *Citrus reticulata* (mandarins and tangerines), *Citrus*
356 *aurantium* (sour orange), tangors or hybrids of sweet orange, and tangerine (Jandrić et al.,
357 2017).

358 Beyond the addition of other fruit juices, one of the artificial ingredients often added is glucose-
359 fructose syrup (Europe) or high fructose corn syrup (United States). These syrups are added as
360 an alternative to sucrose due to their viscosity, which contributes to preventing crystallization
361 and having a lower cost than sucrose (Wójcik & Jakubowska, 2021). Both components are
362 associated with obesity risks when consumed in excess (Yu et al., 2013, Süli et al., 2017).
363 Besides that, corn syrup may have levels of trace mercury resulting from syrup production
364 technology (Wójcik & Jakubowska, 2021).

365 The authenticity of juices is verified by basic analytical information, such as Brix or total
366 acidity, besides biomolecular approaches, and isotopic analysis. Also important is the
367 application of analytical methods that assess the chemical profile of fruit juices including the
368 quantification of sugars, anthocyanins, organic acids, carotenoids, and amino acids. Although
369 these options are usually applied, there is a need for fast and accurate analysis methods to
370 determine the presence of possible adulterants in fruit juices (Wójcik & Jakubowska, 2021).

371 3.2.5. Mycotoxins

372 The mycotoxins are secondary metabolites of mold and fungi, which even in low concentrations
373 are harmful to humans. The main fungi in fruits belong to the genera *Aspergillus*, *Penicillium*
374 and *Alternariae* and give rise to a wide range of mycotoxins, including aflatoxins, produced by
375 *Aspergillus*, ochratoxins produced by *Aspergillus* and *Penicillium*, while citrinin and patulin
376 are mycotoxins produced by *Penicillium*. And finally, *Alternaria* toxins are produced by
377 *Alternariae* fungi (Guo et al., 2021).

378 Mycotoxins can be transferred from the fruit to the juice if the spoiled fruit is not discarded
379 during the beverage making process. Therefore, quality control of the raw material (fruits) is
380 essential to prevent mycotoxin contamination in fruit juices (Gil-Serna & Patiño, 2020).
381 Prevention strategies, both during agricultural production and in beverage production, have
382 proven to be good alternatives to inhibit mycotoxin biosynthesis, including care during the

383 harvest, such as field management, use of biological and chemical agents, types of residence,
384 and post-harvest care, which include improved drying, decontamination processes and care with
385 storage conditions (Mostafidi et al., 2020). It is important to emphasize that, the occurrence of
386 mycotoxins depends on several factors, including, the composition of the food matrix, moisture
387 content, temperature, pH, relative humidity, and physical damage (Pallarés et al., 2021).

388 The great concern with mycotoxin contamination is related to adverse health effects since
389 chronic exposure to these substances can result in neurotoxic, immunological, mutagenic,
390 genotoxic, carcinogenic, and teratogenic problems (Guo et al., 2021, Marin et al., 2013). The
391 Codex Alimentarius, the European Union, and countries such as the United States, Canada, and
392 China established maximum levels for some mycotoxins in fruit juices, with a maximum of 50
393 $\mu\text{g kg}^{-1}$ for patulin and 2 $\mu\text{g kg}^{-1}$ for ochratoxin A (Guo et al., 2021).

394 3.2.6. Bisphenols

395 Bisphenols are a class of anthropogenic chemical substances widely used as modifier
396 monomers in plastic production to improve material properties, including greater flexibility and
397 strength (Hafez et al., 2019). A total of seventeen bisphenols have been documented for
398 industrial applications, including bisphenol A, bisphenol B, bisphenol F, bisphenol AF and
399 tetrabromobisphenol A. Among them, bisphenol A (BPA) is the most widely applied in plastic
400 production, including the production of food packaging and beverage packaging. Due to its low
401 production cost, high thermal and chemical stability, BPA is widely applied as a raw material
402 (Khan et al., 2021).

403 Incomplete polymerization processes or polymer degradation can easily result in the migration
404 of bisphenols from packaging to food and beverages during prolonged storage and at elevated
405 temperatures (D. Yang et al., 2018). Exposure to these compounds poses a potential risk to
406 human health since bisphenols are classified as endocrine disruptors, with a negative effect on
407 the hormonal system. Furthermore, studies indicate that BPA can cause diseases related to the
408 cardiovascular, metabolic, and immune systems, as well as diabetes (Hafez et al., 2019).

409 3.2.7. Pollutants

410 Fruit juices can contain trace-level contaminants belonging to different classes of organic
411 pollutants, including polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons
412 (PAHs). The PCBs are a group of synthetic organic compounds considered to be persistent
413 organic pollutants in the environment. They are highly lipophilic compounds and for this
414 reason, they are found in concentrations in the range of ng mL^{-1} in aqueous samples, such as
415 fruit juices (Abujaber et al., 2019). Exposure to PCBs is associated with adverse
416 neurobehavioral problems, endocrine disturbances, and immunological effects (Darvishnejad
417 & Ebrahimzadeh, 2019).

418 The PAHs, in turn, constitute one of the largest groups of contaminants present in different
419 matrices, including food. These compounds are derived from the incomplete combustion of
420 organic matter and can come from natural and anthropogenic processes such as pollution, food
421 processing, packaging, and thermal procedures such as cooking. PAHs are often found in

422 different beverages, including fruit juices. The great concern for the scientific community and
423 the food industry is related to harmful health properties, including carcinogenic and mutagenic
424 activities (Rascón et al., 2018).

425 **4. Application of analytical techniques**

426 Food is a complex heterogeneous mixture of a wide range of chemical constituents as well as a
427 wide array of additives and contaminants. Product composition analysis is a prerequisite for
428 verification of product quality, fulfilment of regulatory enforcements, checking compliance
429 with national and international food standards, contracting specifications and nutrient labelling
430 requirements and providing quality assurance for the use of the product for the supplementation
431 of other foods (Kumar & Gowda, 2014). These aspects also apply to the berry fruit which
432 intends to be the sub-product for the production of juice. It also must be noted that even though
433 fruit juices are generally considered healthy, there are many risks associated with mishandling
434 both fruits and juices themselves (Abisso et al., 2018).

435 As was previously mentioned, it is important to collect information on both, the positive and
436 negative effects connected with berry fruits and juices. While some of risks are minimized in
437 the case of commercially available juices, quality assessment is of particular importance in the
438 case of homemade juices, since consumers are not bound to obey the same sanitary standards
439 as food producers (Li et al., 2017). And here, analytical chemists are coming with the specific
440 practical knowledge of how to use analytical techniques for quality control of berry fruit juice.
441 In this chapter, the specific techniques are described with examples of their application in the
442 analysis of berry fruit juice. The focus is mainly placed on the novel methods for fruit juices
443 quality assessment in the context of green analytical chemistry, but also we give diverse and
444 interesting examples of studies that show the possibilities of future developments.

445 4.1. Benefits assessment

446 4.1. 1. Spectroscopic techniques

447 Spectroscopic techniques are a useful analytical platform for any food screening because their
448 application is rapid, mobile, and, in the case of some techniques, non-destructive. Spectroscopy
449 is a popular technique commonly used at the stage of preliminary research to determine the
450 summary parameters (Boqué & Giussani, 2021).

451 The spectrophotometric technique was applied for the evaluation of the effect of total phenolic
452 concentration on the flavor of blue berry juice. The total phenolic content of blueberry juices
453 from different cultivars was determined using the Folin-Ciocalteu method. The method showed
454 good sensitivity (0.05-0.5g/L) (Bett-Garber et al., 2015). The Folin-Ciocalteu method is widely
455 applied for the analysis of total phenolic compounds, however, this method has limitations, as
456 the Folin-Ciocalteu reagent is not specific for phenolic compounds and may also react with
457 other oxidizable compounds present in the sample, including ascorbic acid, amino acids, sugars,
458 ferrous ions, among others, thus, the total polyphenol content can be overestimated (Granato et
459 al., 2016). Furthermore, the Folin-Ciocalteu method does not allow the quantification of
460 individual phenolic compounds. Therefore, it is not possible to correlate specific compounds
461 and their individual properties (Martins et al., 2022). Bett-Garber et al. (2015) related sensory

462 analyzes together with physical-chemical analyzes and concluded that berries from different
463 cultivars showed variability in their aroma and flavor. However, it was noticed that polyphenols
464 had no significant effect on the bitter and astringent taste of berries but higher polyphenols
465 concentrations contributed to more intense sweet taste (Bett-Garber et al., 2015). This work
466 presents that the impact of juice composition on flavor is very complicated, and in fact, the
467 estimating flavor with physicochemical parameters is a difficult task due to the composition of
468 the juice. In addition to the phenolic composition, other factors can affect the flavor of fruits
469 and juices, including natural sugars and organic acids. Furthermore, bitterness can be especially
470 influenced by the presence of iridoids. Iridoids are monoterpenoids synthesized naturally in
471 different plants and are characterized by a very bitter taste. However, it should be noted that
472 iridoids present in plants have diverse biological activities, such as anti-inflammatory,
473 antioxidative, anti-cancer, etc (Oszmiański & Kucharska, 2018).

474 In the work presented by Tolić et al. (2017), effects of weather conditions on fruit quality
475 attributes, phenolic compounds and antioxidant capacity of selected chokeberry juice over three
476 consecutive years were investigated. Total phenols were determined by Folin-Ciocalteu
477 method, while the pH differential spectroscopic method was used for total anthocyanins
478 determination. Although quality parameters and phenolic composition vary over growing
479 seasons, chokeberry juices from all three seasons have very high contents of phenolic
480 substances and high values of antioxidant properties. This allows to state that weather
481 conditions affect the concentration of antioxidative compounds. In addition, the results
482 presented in this study showed that chokeberry juices characterized by high phenolic compound
483 values had also high antioxidant activity. This is why it can be deduced that due to the high
484 proportion of natural antioxidants their consumption could bring health benefits.

485 As the measurement of total anthocyanin value along with polymeric colour can be very useful
486 to assess the quality of colour of anthocyanin-containing juices during heating, the knowledge
487 on kinetic of anthocyanins degradation in specific temperature is required. Such research was
488 performed by Danişman et al. (2016). The kinetic degradation of anthocyanins in grape juices
489 was studied in the temperature range of 70-90°C. The absorbance of diluted
490 grape juice samples in buffers at pH 1.0 and 4.5 were measured at 520 nm (λ_{max}) and 700 nm
491 using an UV-Vis spectrophotometer. The method had simple sample preparation steps and an
492 acceptable LOD value. In addition, the formation kinetics of percent polymeric colour (%PC)
493 was also studied by application of the bisulphite bleaching method. High correlations were
494 found between anthocyanin degradation and % PC formation during heating. The obtained
495 results allow to state that due to the fact that the heat treatment had a significant effect on
496 monomeric anthocyanins and polymeric colour, it should be carefully optimised to decrease the
497 anthocyanin losses and polymeric colour formation in the commercial processing of
498 grapes into juice.

499 4.1.2. Chromatographic techniques

500 Many properties as well as content of specific important compounds can be determine by
501 application of different chromatographic methods. Such analysis allow to estimate and evaluate
502 the quality of different berries. Furthermore, the application of chromatographic analyses is

503 recommended for identification and quantification of specific compounds, allowing the correct
504 correlation of such compounds and their properties. In this chapter, the specific examples of
505 such applications are presented.

506 As was previously mentioned berries are commonly consumed as juice, however, the juice-
507 processing conditions could affect their bioactive compounds. This is why many researches are
508 published to present the impact of pasteurization conditions on the bioactive compounds
509 content. The effect of thermal treatment on the phenolic compounds, anthocyanins and
510 ellagitannins content as well as the antioxidant capacity of black berry was evaluated by the
511 HPLC-PAD method (Azofeifa et al., 2015). With the use of the described method, it was
512 possible to follow the concentration of phenolic compounds, anthocyanins and ellagitannins
513 content. Neither of the two pasteurization conditions that were examined in this study
514 significantly altered the concentrations of the total polyphenols or total/individual ellagitannins
515 compared to those in the non-pasteurized juice. On the other hand, the concentration of
516 anthocyanins significantly decreased. Over and above, the pasteurized juice was found to
517 inhibit the peroxidation as well as non-pasteurized juice.

518 Another chromatographic method was used for analysis and comparison of the phenolic
519 composition, anthocyanins, and antioxidant capacity in blackcurrant juice from ten different
520 cultivars (Kowalski & Gonzalez de Mejia, 2021). In this work, the ultra-high performance liquid
521 chromatography (UHPLC) method was used after sample extraction from freeze-dried black
522 currant juice with methanol acidified with acetic acid. This method has the main advantage over
523 the HPLC method of having shorter analysis time and hence, less solvents consumption
524 (Azofeifa et al., 2015). It was found that anthocyanins content varied in the collected samples,
525 moreover, anthocyanins were found predominantly in the skins of the fruit. In general, the
526 findings of this study clearly indicate that the juices obtained from different blackcurrant
527 cultivars differ with respect to a number of characteristics of interest.

528 In another work, folate vitamers and total folate in different berries were estimated using
529 UHPLC-MS/MS (Zou et al., 2019). In addition, the changes in their concentration during
530 handling of berries into juice were examined. In this method, a simple extraction method
531 (boiling and centrifugation) was used followed by solid-phase extraction for further
532 purification. According to the obtained results, the overall folate yield in the juicing fractions
533 differed amongst berries (strawberries and blackberries had the highest total folate values (93–
534 118 g/100g), whereas blueberries had the lowest total folate contents). The total folates in all
535 tested were raised by 7 to 12 % after juicing which may be due to excessive release of folates
536 from the fruit matrix during processing. In general, it can be concluded, that most of the
537 investigated berries are good to excellent folate sources.

538 In case of red fruit juices, a selected method for quality and authentication is International
539 Federation of Fruit-Juice Producers (IFU) Method No. 71 (1998), which allows to determine
540 anthocyanin profiles by HPLC with visible detection. Despite the fact that the principle of the
541 method is simple and the specific compounds of red fruit juice matrices or adulterations can be
542 detected, correct interpretations of chromatograms are not as easy as expected (Obón et al.,
543 2011), this is why the method is many often modified. Such modification was applied in the

544 research performed in order to determine the composition of selected red fruit and vegetable
545 juices and to evaluate their quality and authenticity. Profiles of anthocyanins, betacyanins,
546 synthetic red pigments, hydroxycinnamic acids, hydroxybenzoic acids and catechins in these
547 fruits were studied with the use of the HPLC-UV-VIS or HPLC-fluorescence detector methods
548 (Obón et al., 2011). The method succeeded to separate all the studied components in 46 min
549 analysis time, hence it can be considered as a useful technique for quality and authenticity
550 control of fruit based products and for the detection of fraudulent mixtures with synthetic or
551 natural red food pigments. On the other hand, as health claims of the red drinks can be related
552 to their polyphenol contents, this method could be applied within the juice industry to label the
553 content of components with potential health benefits.

554 The outcome of enzymatic processing on blackcurrant juices was studied through analysis of
555 anthocyanins, flavonol glycosides, hydroxycinnamic acids, sugars, and acids content
556 (Laaksonen et al., 2014). Analysis of anthocyanins, flavonol glycosides, hydroxycinnamic acids
557 was done through developing an HPLC/DAD method, while sugars and acids were evaluated
558 by GC/FID after derivatization with trimethylsilyl (TMS) derivatizing agent. The results show
559 that the enzyme-aided juices were more astringent and bitter than the non-enzymatic juices.
560 The reason was connected with lower contents of sugars, higher contents of phenolic
561 compounds, and lower pH and sugar/acid ratio. In general, the non-enzyme-aided juices
562 obtained higher ranking in flavour, while the enzyme-aided juices received more points in odour
563 parameter.

564 In addition to evaluate the content of specific bioactive compounds in berry juices, their stability
565 in higher temperatures is also of high importance, and thus, researchers also are focused on this
566 aspects. The stability of polyphenols (anthocyanins, flavanols and phenolic compounds) in
567 chokeberry and blue berried honeysuckle juices previously subjected to one of two sterilization
568 methods (traditional thermal method and sterilization using Enbiojet® Microwave Flow
569 Pesterizer) was tested and compared. (Piasek et al., 2011). The chemical properties verified
570 included determinations of anthocyanins and other polyphenols by HPLC-DAD-MS, however
571 the profiles of antioxidants were obtained by application post-column derivatization. The
572 concentration of phenolic acids and flavonoids (except anthocyanins) did not change
573 significantly under the influence of microwave-assisted sterilization. Moreover, it was observed
574 that using the EnbioJet device, the decrease in anthocyanin content was lower compared to the
575 conventional thermal method, especially in the case of blue berried honeysuckle juice. The
576 present results allow to state that sterilization with EnbioJet® Microwave Flow Pasteurizer is
577 highly conservative as regards bioactive phytochemicals found in examined berry juices. This
578 conclusion could be true for other plant preparations rich in bioactive phytochemicals.

579 Another aspect of the application of chromatographic techniques in the context of determining
580 compounds that have beneficial effect on human health, is their use to assess authenticity or
581 possible adulteration of selected berry juices. In the work (Zhang et al., 2018), a metabolomic
582 approach for authentication of berry fruit juices by liquid chromatography coupled with
583 quadrupole time-of-flight mass spectrometry (LC-QTOF-MS) was established. In the
584 untargeted metabolomics analysis, obtained data were subjected to chemometric analysis such
585 as principal component analysis-discriminant analysis. In the targeted metabolomics analysis,

586 the 41 juice biomarkers, such as flavonoids and anthocyanins provided to separate adulterated
587 juices from berry fruit juices. In addition, adulterants have different flavonoid glycosylation
588 patterns as well as noteworthy differences in phenolic acids. One can conclude that the
589 introduced LC-QTOF-MS-based metabolomic method can be used as a powerful tool to verify
590 the quality of berry fruit juices.

591 In addition, the use of HPLC system in determination of specific parameters, ion
592 chromatography (IC) is also an option while speaking about cations and anions of selected
593 compounds. Such technique coupled to suppressed conductivity detection was applied for
594 simultaneous analysis of organic acids and inorganic anions in different fruit juices including
595 blueberry and grape juices (Uzhel et al., 2021). In contrast to HPLC, a simple sample pre-
596 treatment method was applied (only filtration through 0.45 Mm membrane filter and dilution).
597 In this method, a novel hyperbranched anion exchanger was synthesized and successfully
598 applied to provide the baseline separation of glycolic, acetic, formic, and lactic acids, which are
599 not resolved to baseline with modern commercially available columns when purely aqueous
600 eluents are used. The most important factor used to improve selectivity of stationary phase was
601 the introduction of dicarboxylic aspartic acid into the internal part of positively charged
602 hyperbranched phase. This solution allowed to separate the selected organic acids applying
603 KOH as an eluent without adding traditional, organic solvents. The study show, that the main
604 organic acid present in blueberry juice was found to be citric acid (77-87% of total acids
605 content), which improves ketosis and prevents diabetes, followed by malic acid (protects from
606 ischemic lesions and has a positive effect on myocardium) and quinic acids (4-11% of total
607 acids), which can be metabolized to hippuric acid, which is a strong antibacterial agent. This
608 novel ion exchanger can be used for the estimation of organic acid profiles in food quality
609 control to detect its deterioration during storage or authenticity assessment.

610 4.2. Risk assessment

611 4.2.1. Spectroscopic techniques

612 In most of the published articles dedicated to the issue of metal content in fruit juices, an atomic
613 absorption spectroscopy (AAS) (Abbasi et al., 2020; Anastácio et al., 2018; Okhravi et al.,
614 2020; Sorouraddin et al., 2020) and atomic emission spectroscopy (AES) (Demir et al., 2020)
615 were applied with different sample extraction methods.

616 Some researchers performed, a microwave-assisted digestion with either the combination of
617 nitric acid and hydrogen peroxide (Anastácio et al., 2018) or nitric acid and perchloric acid
618 (Abbasi et al., 2020) followed by analysis using AAS. Microwave-assisted digestion results in
619 shorter digestion time and avoids loss of metals by volatilization. However, the latter showed
620 lower sensitivity.

621 Moreover, Co and Ni were analysed in pomegranate juice with the application of a graphite
622 furnace atomic absorption spectrometer after complexation with 8-hydroxyquinoline and
623 liquid-liquid microextraction (Okhravi et al., 2020). The most outstanding advantage of a given
624 technique was the use of nitrogen instead of toxic chlorinated solvents. The extraction was
625 performed within seconds and LODs on the level of 0.36 µg/L for Ni(II) and 0.20 µg/L for

626 Co(II) were achieved. Another study shows, a green deep eutectic solvent dispersive liquid
627 liquid extraction method (DLLE) used to extract and preconcentrate metals from the grape
628 juice samples (Sorouraddin et al., 2020) followed by their analysis using FAAS. The most
629 important factor in the DLLE technique is the choice of a relevant extraction and dispersion
630 solvent. Hence, extraction solvent must have a high affinity for analytes, low solubility, high
631 sample stability, be a liquid under standard conditions and have low vapor pressure (Makoś et
632 al., 2020). In the study, the deep eutectic solvent acted as both a complexing agent and an
633 extraction solvent. Application of deep eutectic solvents instead of hazardous chlorinated
634 organic solvents is more and more popular. It may be due to their physicochemical properties:
635 viscosity, density, acidity, basicity, polarity and good extractability. They can be designed
636 according to the needs. Moreover, they are biodegradable, non-flammable. Thus, their use is in
637 line with the requirements of green analytical chemistry (Makoś et al., 2020).

638 Several researches intended to assess the quality of fruit juices, indicated the presence of some
639 metals like Cr, Ni, Mn above their permissible limits established by Decree-Law 306/2007 from
640 27th August of Portuguese Legislation for drinking water, WHO (World Health Organization)
641 and USEPA (United States Environmental Protection Agency)(Abbasi et al., 2020; Anastácio
642 et al., 2018; Sorouraddin et al., 2020). Moreover, the health risks index (HRI) for some metals
643 was evaluated. HRI was calculated as a proportion between the estimated daily intake of the
644 metal and reference oral dose for each metal and the body weight. When HRI is below 1, the
645 exposure to metal is considered as safe. However, the results showed HRI for Cd, Cr and Pb
646 over 1, what signalize a danger for human health. Thus, the data confirms the importance of
647 monitoring of metal ions in fruit juices. (Abbasi et al., 2020)

648 Another approach of metal determination in fruit juices and nectars were done by inductively
649 coupled plasma optical emission spectrometry (ICP-OES) method after microwave-assisted
650 digestion (Demir et al., 2020). This method is characterized by fast extraction without the need
651 of using organic solvents. Additionally, it showed a high sensitivity for all analyzed metals.

652 4.2.2. Chromatographic techniques

653 Chromatographic techniques including GC and HPLC are widely applied techniques when it
654 comes to monitor varietal organic contaminations in juice samples, as shown in Table 1.
655 *Alternaria* mycotoxins in pomegranate fruit and juice samples were determined with the use of
656 HPLC-DAD method (Myresiotis et al., 2015). In this method, samples were subjected to the
657 QuEChERS-based extraction method using acetonitrile (ACN) as organic solvent. Moreover,
658 ACN was also used as the organic modifier in the applied mobile phase what is a big drawback.
659 Hence the single analysis lasts 35 min the ACN is consumed in large quantities per each sample.
660 On the other hand, high sensitivity being able to detect at targeted analysis even trace amount
661 of toxins (LODs <0.02 µg/mL) was achieved. PAHs is another group of compounds being a
662 subject of the study when quality of berry fruit juices was discussed. Analysis of a given group
663 of compounds is very challenging, because they are present in very low concentration, so they
664 need a pre-concentration step as well as a very sensitive analytical method of analysis (Zhao et
665 al., 2009). Analysis of eight PAHs in grape juice samples was performed by using dispersive
666 liquid liquid microextraction coupled with high performance liquid chromatography with

667 fluorescence detection (DLLME-HPLC-FLD). DLLME was based on ACN (as a dispersive
668 solvent) and methylene chloride (as an extraction solvent). Despite using toxic organic solvents
669 in sample extraction, this method of extraction had a high enrichment factor (enrichment factors
670 ranged from 296 to 462) leading to a wide linear range and high sensitivity as well as low
671 detection limit.

672 Another method was published for extraction and pre-concentration of twelve PAHs depending
673 on using a vortex-assisted dispersive solid-phase microextraction (VA-d- μ -SPME) using ionic
674 liquid-modified metal-organic frameworks (ILMIL-100(Fe)) followed by GC/FID
675 (Nasrollahpour et al., 2017). This method has some advantages over the DLLME (Zhao et al.,
676 2009), such as shorter extraction time (only one minute) and higher extraction efficiency (due
677 to the use of ILMIL-100(Fe)). Combination of both (i.e., ionic liquid and MOF) lead to higher
678 sorbent capacity. The developed GC method had high sensitivity (linearity range 0.02-200
679 ng/mL) and a short analysis time (15 minutes).

680 Other researchers proposed GC/FID method for determination of six organic esters. In this
681 method, sample pretreatment and pre-concentration were performed using polycarbazole/ionic
682 liquid fiber for HS-PME. The synthesized fiber was cheap and had a long lifetime. Moreover,
683 the extraction method showed high efficiency, however the extraction time was equal to 40
684 minutes. The developed GC method showed a wide linear range, as shown in Table.1. Total
685 time of the analysis was 26 min (Feng et al., 2015).

686 Furthermore, a highly sensitive GC-MS/MS method was published for analysis of twelve
687 phthalic acid esters in grape juice (Rodríguez-Ramos et al., 2020). In this method, extraction
688 and pre-concentration of the target esters were carried out by a modified QuEChERS method.
689 Results of the validation of the extraction method showed high extraction recovery (75–115%)
690 and good repeatability. The high sensitivity of the developed method facilitated its application
691 for analysis of the cited analytes in grape juice samples and the results confirmed the presence
692 of some of the studied esters at different concentration levels in some of the tested samples.

693 As was previously mentioned, BAs are a group of compounds that are important to be
694 monitored due to many reasons. As the BAs are usually hydrophobic, poor chromophores, and
695 their concentration is usually low in complicated matrices, their determination in food samples
696 and beverages is a challenging analytical task. Chemical derivatization by different reagents
697 like dansyl chloride (for both primary and secondary amines) (Gomez-Gomez et al., 2018;
698 Saaid et al., 2009) and O-phthaldialdehyde (specific for primary amines) is commonly used to
699 improve methods sensitivity (Kelly et al., 2010).

700 BAs in different juice samples were analyzed by HPLC methods after different sample
701 pretreatment and derivatization (Gomez-Gomez et al., 2018; Kelly et al., 2010; Saaid et al.,
702 2009). In the method presented by Gomez-Gomez et al. (2018), BAs and phenolic compounds
703 in grape juice were analysed to evaluate their functional and nutritional quality. Samples were
704 homogenized with perchloric acid followed by derivatization with dansyl chloride. As well as,
705 liquid-liquid extraction with toluene was performed. Analysis of eight BAs by HPLC-UV with
706 a mobile phases (A) 100% ACN and (B) 50% ACN was done within 25 minutes. While,
707 phenolic compounds were analysed with the use of the UPLC-UV method applying mobile

708 phases of aqueous phosphoric acid (0.85%) and ACN (100%). Principle component analysis
709 was then carried out and results showed that a higher phenolic compound content may be linked
710 to a higher BAs content. The discovered association also showed that some bacteria that
711 synthesize BAs are becoming more active at high pH levels. Some microbes' metabolism is
712 inhibited by low pH, which prevents the synthesis of BAs (Gomez-Gomez et al., 2018). HPLC-
713 UV method was also applied to determine BAs in blackcurrant and red grape juices after
714 dilution in 0.1M HCl and aqueous extraction followed by derivatization with dansyl chloride
715 (Saaid et al., 2009). The method had a wide linear range (Table 1), acceptable detection and
716 quantitation limits as well as recoveries in the range between 90.0 and 106.3%. Seven BAs in
717 grape juice was also evaluated after automated in-loop pre-column derivatization with an O-
718 phthaldialdehyde and N-acetyl-L-cysteine, followed by HPLC analysis with fluorescence
719 detection (Kelly et al., 2010). Chromatographic analysis takes 39 minutes. Because of the
720 method's great sensitivity, no sample preparation other than a straightforward dilution was
721 needed prior to derivatization, eliminating the necessity for an internal standard.

722 In recent years, the application of lactic acid bacteria (LAB) inoculation to fruit and juices
723 processing has gained popularity in the production of unique non-alcoholic fermented
724 beverages. It is a simple and valuable biotechnological method that allows fruits to be processed
725 into products with a longer shelf life. The effect of LAB inoculation on the chemical
726 composition of bog bilberry juice was studied using an HPLC method (Chen et al., 2019).
727 ACN:methanol in the ratio 4:1 (phase A) and 25 mM acetate buffer mixture were used as a
728 mobile phase and separation of seven BAs within 93 minutes. The study also involved the effect
729 of LAB on reducing sugars, organic acids, anthocyanins, and non-anthocyanins phenolic
730 compounds. Results disclosed that inoculation with LAB resulted in significant changes in the
731 juice composition. Sugars, anthocyanins, total phenolic acids, total flavanols, and amino acids
732 contents decreased in the juices after incubation but no changes in organic acids were noticed.
733 It was also observed, that the content of four biogenic amines as tyramine, cadaverine,
734 putrescine and phenylethylamine decreased after incubation but isoleucine content increased 8
735 times. The findings of this study should be taken into consideration to design a fermentation
736 process that does not result in significant losses of various health-promoting components and
737 does not result in health risk related with the BAs content (Chen et al., 2019).

738 Additionally, a UPLC method was published for testing nine BAs in grape juice to be used as
739 a quality marker for grape-derived products (Gomez et al., 2020). The derivatization process
740 for BAs was done with the use of dansyl chloride, while the mobile phase consisted of a mixture
741 of 100% ACN (phase A) and 50% ACN (phase B). Analysis time was shorter as compared to
742 those obtained using HPLC-based procedures (Chen et al., 2019; Gomez-Gomez et al., 2018;
743 Kelly et al., 2010; Saaid et al., 2009). Additionally, it had a wide linear range and good
744 sensitivity as presented in Table 1.

745 Moreover, two GC-MS/MS methods were applied for the determination of BAs in grape juice
746 samples (Cunha et al., 2011; Fernandes & Ferreira, 2000). Methods differs in the extraction
747 techniques used. In the first one (Fernandes & Ferreira, 2000), the back-extraction with 0.1 M
748 HCl was done after the amines have been extracted with the ion-pairing agent bis-2-
749 ethylhexylphosphate dissolved in chloroform. Derivatization of the extracted amines was

750 performed by using heptafluorobutyric anhydride reagent. Seven amines (β -phenylethylamine,
751 tyramine, 1,3-diaminopropane, putrescine, cadaverine, spermidine and spermine) were
752 quantified using this method with high sensitivity, accuracy and reproducibility. In the second
753 one (Cunha et al., 2011), liquid-liquid extraction was used with a toluene as an extraction
754 solvent and isobutyl chloroformate as an derivatizing agent. Application of a given method
755 enables quantification of 22 biogenic amines in 25 minutes.

756 Pesticides residues are also often present in complex matrices such as berry juice at very small
757 concentrations, hence for detection of these harmful components highly sensitive and selective
758 analytical methods are needed.

759 The most widely used chromatographic technique for pesticide determination in fruit juice
760 samples is GC with the application of different extraction techniques and detectors like:

- 761 • Liquid-liquid microextraction combined with gas chromatography coupled with time-
762 of-flight mass spectrometry (LLME GC-ToFMS) which was developed for screening
763 165 contaminants from the group of pesticides and dioxins like PCBs and PAHs.
764 Despite the satisfactory recoveries (76-120%) and simplicity of the extraction method,
765 the use of chloroform and cyclohexane makes it unfavourable from the Green
766 Analytical Chemistry requirements (Dasgupta et al., 2011).
- 767 • Multiresidue matrix solid-phase dispersion combined with GC coupled with electron
768 capture detector (ECD) and nitrogen-phosphorus detector (NPD). The method allows
769 to determine 160 pesticides in berry fruits and their products. The disadvantage of the
770 proposed approach is the use of hexane as one of the extraction solvent (Wołejko et al.,
771 2014a).
- 772 • Counter current salting-out homogenous liquid-liquid extraction combined with
773 dispersive liquid-liquid microextraction coupled with GC/FID (CCSHLLE-DLLME
774 GC/FID). The approach uses ACN as a coextraction/disperser, 1,2-DBE as an
775 extraction solvent and demonstrates large linear ranges (even $1-10000 \mu\text{g L}^{-1}$) for the
776 target analytes under the optimum extraction conditions (Farajzadeh et al., 2015).
- 777 • Montmorillonite clay intercalated with ionic liquids co-deposited with polythiophene
778 polymer (PTh IL-Mmt) coated electrochemically on SPME coupled to GC/ECD (PTh
779 IL-Mmt SPME GC/ECD). The method allows determination of 5 analytes in 33 min.
780 The imidazolium group in IL, along with the porous surface structure of the fiber, all
781 contribute to the hybrid material's strong electrostatic contacts, hydrogen bonds, and π -
782 π interactions, which results in a high capacity for adsorption of volatile pesticides
783 (Pelit et al., 2015). The method is characterised by high extraction efficiency (88.7-
784 101.7%), high sensitivity and low detection limit presented in Table 2.
- 785 • Dispersive liquid-liquid extraction coupled with GC and nitrogen – phosphorus
786 detector (DLLME GC/NPD). The method allows to determine three classes of
787 pesticides (triazine, triazole, and neonicotinoid) at the ng mL^{-1} range. It is based on
788 acid-base reaction, in which the extraction solvent (*p*-chloroaniline) is dispersed (by
789 deionized water) into an aqueous sample. In this study low LODs and LOQs and high
790 extraction recoveries and enrichment factors were attained present in Table 2
791 (Farajzadeh et al., 2016).

- 792 • Continuous sample drop flow microextraction combined with GC-MS (CSDF-ME GC-
793 MS) was used to determine phorate, diazinone, dimethoate, disulfoton, and chlorpyrifos
794 from the fruit juice. The application of narrow-necked conical shaped vessel results in
795 short analysis time, high sensitivity and low total solvent consumption (Moinfar et al.,
796 2020).
- 797 • Continuous sample drop flow microextraction combined with GC-MS (CSDF-ME GC-
798 MS) is a similar approach as presented in the year 2020 however, different design of
799 extraction vessel (the extraction vessel was a conical open-end vial set in a little
800 container filled with double-distilled water) and halogen-free organic extraction solvent
801 were applied. Lower limit of LODs and LOQs than in previously published report were
802 achieved and the extraction recoveries in the range between 25.5 and 48.0% (Moinfar
803 et al., 2021).
- 804 • Dispersive liquid-liquid microextraction coupled with GC/FID. And D (DLLME GC-
805 FID) was used to determine pesticide residues including penconazole, chlorpyrifos,
806 ametryn, clodinafop-propargyl, diniconazole, oxadiazon, and fenpropathrin from fruit
807 juice. The *iso*-propanol was used as disperser and 1,2-dibromoethane as an extraction
808 solvent. Moreover special shaped vessel (downward vaporization gas orientation) was
809 designed and used for vaporization and ultra-preconcentration of the extract from
810 DLLME step. The innovations of this study were good outcomes (presented in Table
811 2) using readily available, straightforward equipment. Recoveries from extraction were
812 on the level of 55-89% (Farajzadeh et al., 2021).
- 813 • Multi-plug filtration clean-up combined with gas chromatography-electrostatic field
814 orbitrap high resolution mass spectrometry (m-PFC GC-Orbitrap/MS) is a method
815 developed for screening of 350 pesticides in grape and strawberry juice samples. This
816 extraction method was found to be simple and time-effective. Moreover, highly
817 efficient clean-up of all targeted samples was observed due to the fact that the m-PFC
818 column has the advantage of multiwall carbon nanotubes (MWCNTs). MWCNTs have
819 superior adsorption capacities compared to other sorbents because of their
820 extraordinarily high surface area and distinctive structure. The extraction recovery was
821 found to be 72.8–122.4%, revealing the high performance of the used extraction
822 method (Meng et al., 2021).

823 Another approach used for pesticides determination utilizes LC. Seven insecticides in grape
824 juice samples were analysed using HPLC (M. Yang et al., 2014) after the ionic liquid-assisted
825 LLME, which was based on the solidification of floating organic droplets utilizing a bell-shaped
826 collection device (BSCD). The modification of the traditional LLME method increased its
827 extraction efficiency since the use of BSCD allowed easier collection of the mixed extraction
828 solvents (1-dodecanol and IL, which replaced commonly applied chlorinated solvents) and
829 quicker separation after solidification. The method resulted in an efficient concentration of the
830 studied components in the tested samples, the enrichment factor was in the range of 160 to 246
831 with little consumption of organic solvents.

832 Picó & Kozmutza (2007) developed a highly sensitive LC-MS/MS for the analysis of four
833 pesticides and their metabolites in different grape juice samples (Picó & Kozmutza, 2007).



834 Solid-phase extraction (SPE) was carried out before the analysis, which yield in good extraction
835 recovery (more than 80%), high sensitivity and low quantitation limit, as shown in Table 1.
836 Many of the pesticides are prone to degrade due to oxidative mechanisms, thus authors checked
837 the role of antioxidant for the increase of the durability of certain of the pesticide in fruit juice.
838 Results indicated that the degradation rate of the targeted pesticides was slower in grape juice
839 and quercetine-containing aqueous solutions than in water. These findings suggested that
840 natural antioxidants found in fruit juices might decrease pesticide breakdown rates and enhance
841 their persistence.

842 Timofeeva et al. (2017) developed another fully-automated LC-MS/MS method for the
843 detection of four pesticides in fruits and berry juices. Under the optimized conditions the
844 proposed extraction procedure takes less than 2 min. Apart from that it is simple to perform,
845 inexpensive and does not require complex equipment. However, authors suggest that
846 combination with other pre-concentration method like SPE can improve the sensitivity
847 (Timofeeva et al., 2017).

848 PCBs are another important group of compounds to be monitored in food and beverage samples.
849 Magnetic oleate-coated Fe₃O₄ nanoparticles (OI-coated MNPs) were used for magnetic solid-
850 phase extraction (mSPE) of selected PCBs from grape juice samples followed by GC-MS/MS
851 analysis (Pérez et al., 2016). Authors compared the developed method with other presented in
852 the literature like DLLE-SFO. It was showed that recovery obtained in this research was
853 between 52-85% which was lower than in DLLE-SFO (73-106%). However, the mSPE GC-
854 MS/MS method achieved higher sensitivity (LOD in the range between 1.6-5.4 ng L⁻¹) than in
855 the other work (3.7-18.5 ng L⁻¹). Although no PCB was detected in real samples, the method
856 validation results confirmed its ability to determine targeted chemicals in different samples.

857 Furan and its derivatives are another group of compounds being of high interest of researchers.
858 Furans are heterocyclic compounds, which contribute to the sensory qualities of a wide range
859 of thermally processed foods (Shen et al., 2016). Shen, et al. (2016) proposed to determine
860 furans in the foodstuff including grape, blueberry and pomegranate juice by static headspace
861 GC-MS. Method was characterized by simple sample preparation (only homogenization by
862 manual shaking and sodium chloride addition was needed). Satisfactory validation parameters
863 were achieved for 13 furans determination (Table 1). However, the GC-MS analysis last more
864 than 40 min. Results of this study revealed that furans were detected in trace amounts in the
865 tested fruit juices (Shen et al., 2016).

866

867 On the other hand, the aromatic profile of pomegranate fresh and commercial juices was studied
868 and correlated to their sensory flavors using partial least squares regression (Vázquez-Araújo
869 et al., 2011). In this study, a headspace-SPME combined with the GC-MS method was used.
870 This study showed that there was a significant difference between fresh and commercial juices.
871 Results showed that there were significant changes in their chemical composition with fresh-
872 squeezed juice being distinguished primarily by the presence of terpenes and aldehydes,
873 whereas furans played a key role in commercial juice aroma. Moreover, different juice
874 manufacturing processes were found to alter the aromatic profile of the fresh juice. So,

875 companies should search for different processing methods for pomegranate juice to improve its
876 quality without affecting its health benefits or increasing its health risks.

877

878 Varming, et al. (2004) evaluated the effect on the aroma and thus, the content of aromatic
879 compounds including furans, of blackcurrant juice after the thermal treatment's. For the
880 purpose of the study a headspace GC/MS method (Varming et al., 2004). In this study,
881 blackcurrant juice samples were exposed to different temperatures (45-90°C) for different
882 periods (57, 80, 110, 130 s). Then the aromatic compounds were collected (samples were
883 purged with nitrogen and target compounds were collected into the traps). Collected volatiles
884 were thermally desorbed and determined using GC-MS. The developed analytical method was
885 applied for the determination of 49 aroma compounds involving three furans. Results of the
886 study proved that the concentration of several terpenoids, furans, and phenols have significantly
887 increased after thermal treatment of 90°C for 60 min. However, application of 60°C and less
888 had no influence of the juice aroma compounds composition.

889 Another method used for furans determination was based on the SPME combined with GC-MS
890 designed to distinguish between healthy and noble-rotten grape berries (Furdíková et al., 2019).
891 The concentration of 7 out of 13 significantly differs between healthy and noble-rotten grapes.
892 It was noticed that the content of furans such as: 2-pentylfuran, dihydrofuran-2(3H)-one, 5-
893 butyldihydrofuran-2(3H)-one, 5-pentyldihydrofuran-2(3H)-one, 5-acetyldihydrofuran-2(3H)-
894 one 5-hexyldihydrofuran-2(3H)-one and 5-ethyldihydrofuran-2(3H)-one were higher in noble-
895 rotten grapes than in healthy fruits.

896



897 Table 1 Characterization of analytical methods applied for the metals, mycotoxins, PAHs, aromatic esters, pesticides, biogenic amines and furans determination in different fruit juices.

| methodology | | | | | | | | | | |
|------------------------------|---|---|--------------------|--|---|---|----------------------------|--|-------------------------------|----------------------------|
| Ref | analyte | sample | number of analytes | sample preparation | abbreviation of the analytical technique used | parameters of the technique | time of the analysis [min] | concentration range | LOD | LOQ |
| (Anastácio et al., 2018) | metals | strawberry juice | 5 | microwave-assisted digestion | GFAAS | Detection at 228.8, 357.9, 283.3, 279.5, and 299.44 nm for Cd, Cr, Mn, Pb, and Ni, respectively. | ----- | 2.29-440.09 µg/L | ----- | 0.31-3.65 µg/L |
| (Abbasi et al., 2020b) | metals | red grape juice, Strawberry Jam, Blackcurrant jam, Strawberry canned fruit, cherry canned fruits, | 7 | digestion | FAAS | Detection at 228.8, 240.7, 357.9, 324.8, 248.3, 217, 213.9 nm for Cd, Co, Cr, Cu, Pb, and Zn, respectively. | ----- | 0.08-37.85 mg/kg | 4-10 µg L ⁻¹ | ----- |
| (Okhravi et al., 2020) | metals | pomegranate juice | 2 | liquid nitrogen induced homogenous LLE | FAAS | A Shimadzu AA-6300 FAAS. The radiation sources were cobalt and nickel hollow cathode lamps. Detection wavelengths were 240.7 and 232.0 nm, respectively. Air/acetylene flame with flow rates of 15 and 2.3 L min ⁻¹ , respectively. | ----- | 0.5–20 µg L ⁻¹ for Co 1.0–30 µg L ⁻¹ for Ni | 0.2-0.36 µg L ⁻¹ | 0.5-0.8 µg/L |
| (Sorouraddin et al., 2020) | metals | grape juice | 2 | DES-DLLME | FAAS | A Shimadzu AA-6300 FAAS. The radiation sources were cobalt and nickel hollow cathode lamps. Detection wavelengths were 240.7 and 232.0 nm, respectively. Air/acetylene flame with flow rates of 15 and 2.3 L min ⁻¹ , respectively. | ----- | 0.50-50 µg L ⁻¹ for Co 0.80-50 µg L ⁻¹ for Ni | 0.22-0.30 µg/L | 0.50-0.80 µg/L |
| (Demir et al., 2020) | metals | cherry, pomegranate, grape juice | 21 | microwave-assisted digestion | ICP-OES | Perkin-Elmer Optima 2100 DV ICP-OES . Power of 1.45 kW, plasma flow of 15.0 L min ⁻¹ , the auxiliary flow of 0.8 L min ⁻¹ , and nebulizer flow of 1 L min ⁻¹ . | ----- | 0.004-1080 mg/L | 0.0001-0.0063 mg/L | 0.0005-0.0209 mg/L |
| (Myresiotis et al., 2015) | mycotoxins | pomegranate fruits and juices | 3 | QuEChERS based extraction | HPLC-DAD | Thermo SpectraSYSTEM HPLC-DAD. Stationary phase: Hypersil BDS-C18 column (250 × 4.6 mm, 5 µm). Mobile phase: eluent (A) water with 50 µL L ⁻¹ trifluoroacetic acid and eluent (B) acetonitrile with 50 µL L ⁻¹ trifluoroacetic acid. Flow rate: 1 ml min ⁻¹ Injection volume: 20 µL Elution: gradient program: 90% A and 10% B, reaching 50% B after 25 min and 100% B after 30 min. 100% B was maintained for 1 min. Thereafter the gradient was returned to 10% B in 1 min and allowed to equilibrate for 3 min before the next analysis. Temperature: 40 °C | 35 | 0.05-10 µg mL ⁻¹ | 0.02 µg mL ⁻¹ | <0.066 µg mL ⁻¹ |
| (Zhao et al., 2009) | PAHs | grape juice | 8 | DLLME | LC-FLD | Agilent 1200 LC system equipped with FLD Stationary phase: A Zorbax Eclipse XDB-C18 column (150 × 4.6 mm, 5-µm particle size). Mobile phase: a mixture of methanol-water (75:25, v/v). Flow rate: 0.8 mL min ⁻¹ . Temperature: 40 °C. Detection: Fluorescence detection was carried out as follows: 0–20 min λ _{ex} at 256 nm and λ _{em} at 441 nm, 20–35 min λ _{ex} at 270 nm and λ _{em} at 390 nm, 35–55 min λ _{ex} at 290 nm and λ _{em} at 410 nm. | 55 | 0.01-100 µg L ⁻¹ | 0.001-0.01 µg L ⁻¹ | ----- |
| (Nasrollahpour et al., 2017) | polycyclic aromatic hydrocarbons (PAHs) | grape juice | 12 | VA-d-µ-SPE | GC-FID | A Chrompack CP9001 gas chromatography. Stationary phase: CP-Sil 24CB capillary column (30 m × 0.25 mm ID with 0.25 µm). | 15 | 0.02–200 ng/mL | 2.0-5.5 ng/L | 6.0-16.8 ng/L |



| | | | | | | | | | | |
|----------------------------|----------------------------------|-----------------------------------|-----|---|------------|---|------|------------------------------|---------------------------------|---------------------------------|
| | | | | | | Temperature program: 40 °C hold for 3 min, increasing to 100 °C at 10 °C min ⁻¹ and directly to 180 °C at 20 °C min ⁻¹ then hold for 2 min. Chemical instrument SP-6890 GC-FID Stationary phase: SE-54 capillary column (30 m × 0.25 mm × 0.25 μm) Temperature program: 50°C held for 3 min; then increased to 190°C at the rate of 15°C min ⁻¹ , to 210°C at the rate of 5°C min ⁻¹ and kept 10 min at the final temperature. | 26 | 0.061-500 μg/L | 15.3-61 ng L ⁻¹ | ----- |
| (Feng et al., 2015) | aromatic esters | grape juice | 6 | IL based SPME | GC-FID | | | | | |
| (M. Yang et al., 2014) | pesticides | grape juice | 7 | ILSFOD-LLME | HPLC-UV | Agilent 1200 series HPLC system. Stationary phase: Spursil C18 columns (5 μm, 4.6 × 250 mm, Dikma Limited) Mobile phase: acetonitrile–water(75:25, v/v) The flow rate: 1 mL min ⁻¹ Elution: isocratic. Temperature: 25 °C. Detection: UV at 254 nm | <20 | 0.5-500 μg/L | 0.03-0.28 μg/L | ----- |
| (Farajzadeh et al., 2021) | pesticides | pomegranate, grape juice | 7 | DLLME | GC-FID | Shimadzu 2014 gas chromatograph equipped with FID detector. Carrier gas: He, flow rate 30 mL min ⁻¹ . A Zebtron™ capillary column (30 m × 0.25 mm i.d., film thickness 0.25 μm). Temperature program: 50°C for 3 min, then 300°C at a rate of 18°C min ⁻¹ and maintained for 10 min. | 27 | - | 45-78 ng L ⁻¹ | 149-261 ng L ⁻¹ |
| (Wolejko et al., 2014b) | Pesticides and their metabolites | strawberry, raspberry juice | 160 | MSDP | GC-ECD/NPD | Agilent 7890 GC coupled to ECD/NPD -Stationary phase: HP-5 capillary column (30m × 0.32mm × 0.5 μm film thickness) Carrier gas: He, flow rate 3 mL min ⁻¹ Temperature program: 120 to 190 °C at a rate of 16 °C min ⁻¹ , increased to 230 °C at 8 °C min ⁻¹ and then to 285 °C at 18 °C min ⁻¹ , and remain for 18 min. | 30.5 | - | - | - |
| (Farajzadeh et al., 2016) | pesticides | grape juice | 6 | DES | GC-FID | Shimadzu 2014 GC coupled to FID Stationary phase: RTX-1 capillary column (30 m × 0.25 mm i.d., film thickness 0.25 μm) Carrier gas: He Temperature program: 80 °C hold for 3 min, ramped at 10 °C min ⁻¹ until 300 °C, and hold at 300 °C for 5 min | 30 | 1.4-5000 ng mL ⁻¹ | 0.39- 3.1 ng mL ⁻¹ | 1.4-11 ng mL ⁻¹ |
| (Pelit et al., 2015) | pesticides | grape juice | 5 | PTH IL-Mmt SPME | GC-ECD | Agilent Model 7820A Series equipped with HP ECD detector systems. Stationary phase: DB-5-MS column (30 m × 250 μm I.D. and film thickness 0.25 μm). Carrier gas: He, flow rate: 1.0 mL min ⁻¹ . Temperature program: 50 °C for 5 min increased to 150 °C at a rate of 25 °C min ⁻¹ and increased to 220 °C at a rate of 10 °C min ⁻¹ and increased to 280 °C at a rate of 5 °C min ⁻¹ . | 33 | 0.01-50 ng mL ⁻¹ | 0.002-0.667 ng mL ⁻¹ | 0.025-2.224 ng mL ⁻¹ |
| (Gomez-Gomez et al., 2018) | BAAs | grape juice | 8 | homogenization in perchloric acid (5% v/v) and derivatization with dansyl chloride in acetone | HPLC-UV | HPLC (Ultimate 3000 BioRS, Dionex-Thermo Fisher Scientific Inc) Column: ACE 5 C18 (5 μm, 25 cm × 4.6 mm). Mobile phase: (A) acetonitrile (100%), (B) acetonitrile (50%) Flow rate: 0.7 mL/min. Injection volume: 20 μL Elution: gradient as follow:: 0-2 min, 40% A; 2-4 min, 60% A; 4-8 min, 65% A; 8-12 min, 85% A; 12-15 min, 95% A; 15-21 min, 85% A; 21-22 min, 75% A; 22-25 min, 40% A. | 25 | 0.00-35.25 mg/L | ----- | ----- |
| (Saaid et al., 2009) | BAAs | black currant juice and red grape | 5 | dilution with 0.1M HCl (ten times) and derivatization with dansyl chloride | HPLC-UV | PU-1580 Jasco HPLC and LG-1580-04 Jasco UV/VIS detector Column: Waters Spherisorb 5 μm ODS2 column (250 × 4.5 mm). Mobile phase: acetonitrile: water (67:33, v/v) Flow rate: 1.2 mL min ⁻¹ . Detection: UV at 254 nm. | 30 | 0.1-250 mg L ⁻¹ | 4.43 – 7.34 μg L ⁻¹ | 14.76 -24.45 μg L ⁻¹ |
| (Kelly et al., 2010) | BAAs | grape juice | 7 | dilution, filtration, in-loop derivatization with o-phthalaldehyde | HPLC-FLD | A Hewlett-Packard (Agilent Technologies Massy) 1100 series HPLC instrument and G1321A FLD Column: CIL 250 mm × 3 mm Equisil® | 39 | 0.25–10 mg/L | ----- | 0.05-0.25 mg/L |



| | | | | | | | | | | |
|--------------------------------|------------|---|----|--|------------|--|----|-------------------------------|---------------------------------|--------------------------------|
| | | | | and N-acetyl-l-cysteine | | Mobile phase: (A) 95% 0.05 M sodium acetate buffer, pH 6.5 and 5% methanol, (B) methanol-acetonitrile 70–30. Flow rate: 0.5 mL/min. Elution: gradient as follow: 0 min, 3%B; 0-4.5min, 5%B; 4.5-10min, 19%B; 10-16min, 27%B; 16-20min, 42%B; 20-25min, 48%B; 25-32min, 60%B, 32-35min, 3%B. Detection: fluorescence detection at excitation and emission wavelengths of 330 nm and 440 nm, respectively. Temperature: 25 °C | | | | |
| (Chen et al., 2019) | BAs | bog bilberry juice | 7 | sonication, heating at 70 °C for 2 h, cooling down to room temperature and filtering | HPLC-UV | Shimadzu LC-20AT LC system Column: A Venusil XSB C18 column (4.6 × 250 mm, 5 µm (Shimadzu, Japan). Mobile phase: (A) acetonitrile:methanol (4:1, v/v), (B) 25 mM acetate buffer (0.02% sodium azide, pH 5.8). Injection volume: 20 µL Flow rate: 0.9 mL/min. Elution: gradient as follow: 0–20 min, 90%B isocratic; 20–30.5 min, 90%B to 83%B; 30.5–33.5 min, 83%B isocratic; 33.5–65 min, 83%B to 73%B; 65–73 min, 73%B to 28%B; 73–78 min, 28%B to 18%B; 78–82 min, 18%B to 0%B; 82–85 min, 0%B isocratic; 85–90 min, 0%B to 90%B; and 90–93 min, 90%B isocratic. | 93 | 0.01-7.94 mg/L | ----- | ----- |
| (Gomez et al., 2020) | BAs | grape juice | 9 | homogenization, centrifugation, derivatization with dansyl chloride | UPLC-UV | Agilent 1200 Series Rapid Resolution LC system Stationary phase: Agilent Zorbax Eclipse XDB – C18 column (50 mm × 4.6 mm ID, 1.8 µm particle size). Flow rate: 1.0 mL/min Injection volume: 5 µL Temperature: 25 °C Detection: 225 nm. Mobile phase: (A) acetonitrile (100%), (B) acetonitrile (50%) Elution: gradient as follows: 0–2 min, A 40%, B 60%, 2–3 min, A 40–80%, B 60–20%, 3–4 min, A 80–90%, B 20–10%, 4–6 min, A 90–95%, B 10–5%, 6–7 min, A 95–40%, B 5–60%, 7–12 min, A 40%, B 60%. | 12 | 2–150 mg/kg | 0.032-0.098 µg L ⁻¹ | 0.11-0.32 µg L ⁻¹ |
| (Rodríguez-Ramos et al., 2020) | phthalates | grape juice | 11 | QuEChERS extraction | GC-MS/MS | Agilent 7890B GC system coupled to Agilent 7000C MS Stationary phase: HP-5 ms capillary column (15 m × 0.25 mm, 0.25 µm film thickness) Carrier gas: He; flow rate: 1.5 mL min ⁻¹ and 1.7 mL min ⁻¹ for backflush. Temperature program: 70 °C for 2 min. Then, 200 °C at a rate of 25 °C min ⁻¹ and then increased to 260 °C at a rate of 3 °C min ⁻¹ . Finally, the temperature reached 300 °C at a rate of 30 °C min ⁻¹ hold for 4 min. | 33 | 0.5-250 µg L ⁻¹ | ----- | 0.034–1.415 µg L ⁻¹ |
| (Pelit et al., 2015) | pesticides | Gooseberry, blackcurrant, redcurrant, raspberry, strawberry, and the concentrated juice of blackcurrant, redcurrant, raspberry, and strawberry. | 5 | PTh IL-Mmt SPE | GC-ECD | Agilent 7820A Series gas chromatograph equipped with HP ECD detector system. HP-5 capillary column (30 m × 0.32 mm, 0.5 µm film thickness) was used. Carrier gas: He, flow rate: 3.0 mL min ⁻¹ . Temperature program: 50° C/5 min and then 150 at a rate of 25° C min ⁻¹ , increased to 220° C at 10° C min ⁻¹ and then to 280° C at 5° C min ⁻¹ , and remain for 18 min. | 33 | 0.04-0.51 ng mL ⁻¹ | 0.002-0.667 ng mL ⁻¹ | 0.002-2.22 ng mL ⁻¹ |
| (Timofeeva et al., 2017) | pesticides | raspberry juice, cherry juice | 4 | IS-SULLE | HPLC-MS/MS | Shimadzu HPLC-MS/MS system LCMS-8030 Triple Quadrupole Liquid Chromatograph Mass Spectrometer Zorbax Bonus-RP column (100 × 2.1 mm, 3.5 µm). Mobile phase: A - deionized water; B - methanol with 0.1% (v/v) formic acid Flow rate: 0.3 mL min ⁻¹ Elution: gradient elution as followed: 0 – 8 min, 20 – 80 % B; 8 – 11 min, 80 % B. | 11 | 0.01-10 mg L ⁻¹ | 0.0003-0.03 mg L ⁻¹ | ----- |
| (Farajzadeh et al., 2015) | pesticides | grape, sour cherry juices | 11 | CCSHLLE-DLLME | GC-FID | Shimadzu 2014 gas chromatograph. CP-Sil 8CB capillary column (30 m X 0.25 mm i.d. 0.25 µm film thickness) Temperature program: 80°C/3 min and then increased to 300 °C at a | 27 | 0.1–5 µg L ⁻¹ | 0.34-5 µg/L | 1-16 µg/L |

| | | | | | | | | | | |
|--------------------------------------|---------------------------|--|-----|----------------------------------|-----------------|---|-------|-------------------------------|-------------------------------|-------------------------------|
| | | | | | | rate of 8 °C min ⁻¹ , and then maintained at 300 °C and remain for 10 min. | | | | |
| (Farajzadeh & Afshar Mogaddam, 2016) | pesticides | cherry, grape, strawberry juice | 17 | Acid-base DLLME | GC-NPD | GC-1000 gas chromatograph with GLAIND-2200 hydrogen generator (H flow rate 5 mL min ⁻¹). HP-5 MS capillary column (30 m × 0.25 mm i.d.). Temperature program: 80 °C hold for 3 min and then increased to 300°C at a rate of 8 °C min ⁻¹ , and then maintained at 300°C and remain for 10 min. The NPD temperature was maintained at 300 °C. | 40 | 0.1-33 ng mL ⁻¹ | 0.05-0.43 ng mL ⁻¹ | 0.17-1.43 ng mL ⁻¹ |
| (Moinfar et al., 2020) | pesticides | grape juice | 5 | CSDF-ME | GC-MS | Clarus 580 GC equipped with Clarus SQ 85 quadrupole MS system. Carrier gas: He, flow rate of 1.0 mL min ⁻¹ . HP-5MS (30 m × 0.25 mm id, , 0.25-µm film thickness) capillary column. Temperature program: 110°C hold for 0.5 min, then increased to 195 °C with a rate of 20°C min ⁻¹ and hold for 1.5 min. Next, the temperature was increased to 230 °C with a rate of 25 °C min ⁻¹ and hold for 3.5 min. | 10 | 380- 500.0 µg L ⁻¹ | 0.03-1.0 µg L ⁻¹ | 2.0-5.0 µg L ⁻¹ |
| (Moinfar et al., 2021) | pesticides | grape juice | 5 | CSDF-ME | GC-MS | GC-MS, Clarus 580 gas chromatography HP-5MS (30 m, 0.25-µm film thickness × 0.25 mm id) capillary column. Carrier gas: Helium, flow rate: 1.0 mL min ⁻¹ . Temperature programming: The oven temperature of GC was programmed for 0.5 min at 110 °C for the initial hold, then the temperature was raised by 20 °C min ⁻¹ to 195 °C and held for 1.5 min, then heated to 230 °C at 25 °C min ⁻¹ and kept at the same temperature for 3.5 min. | 10 | 1-1.2 µg L ⁻¹ | 0.02-.030 µg L ⁻¹ | 0.07-1.0 µg L ⁻¹ |
| (Meng et al., 2021) | pesticides | grape juice and strawberry juice | 350 | m-PFC | GC-Orbitrap/MS | GC-Orbitrap system Thermo Scientific TG-5MS (30 m × 0.25 mm ID, 0.25 µm) column Carrier gas: He, flow rate: 1.0 mL min ⁻¹ . Temperature program: 40 °C hold 1.5 min then increased to 90 °C at the rate of 25 °C min ⁻¹ , then increased to 180 °C at the rate of 25 °C min ⁻¹ , then increased to 280 °C at the rate of 5 °C min ⁻¹ , then increased to 310 °C at the rate of 10 °C min ⁻¹ , and held at this final temperature for 3 min. | 34 | 5 to 500 µg kg ⁻¹ | 0.3–3.0 µg kg ⁻¹ | 1.0–10.0 µg kg ⁻¹ |
| (Dasgupta et al., 2011) | pesticides, PCBs and PAHs | grape juice, pomegranate juice | 165 | LLME | GC-ToFMS | Pegasus 4D GC-ToFMS system Rtx®-5 capillary column (10 m × 0.18 mm, 0.20 µm) connected in series to a Varian VF-17 ms (1 m × 0.10 mm, 0.10 µm) Carrier gas: ultra-pure grade He. Temperature program: 100 °C hold for 2 min, increased to 200 °C at the rate of 20 °C min ⁻¹ hold for 2 min hold and finally to 285 °C at 20 °C min ⁻¹ hold for 2 min. The secondary oven temperature was consistently set at 10 °C higher than the primary oven. | 15.25 | 1-500 µg L ⁻¹ | 1-250 ng L ⁻¹ | 0.4-1000 ng mL ⁻¹ |
| (Pérez et al., 2016) | PCBs | grape juice | 7 | mSPE | GC-MS/MS | Agilent 7890A GC coupled with Agilent 7000 MS/MS Carrier gas: He, flow rate: 1 mL min ⁻¹ Temperature program: 150 °C hold for 1 min, then increased at 10 °C min ⁻¹ to 280 °C hold for 10 min.. | 15 | 7.5-90 ng mL ⁻¹ | 1.6-2.9 ng L ⁻¹ | 5.2-9.8 ng L ⁻¹ |
| (Shen et al., 2016) | furan and 2-alkylfurans | grape juice, blueberry juice, pomegranate juice. | 8 | homogenization and NaCl addition | GC-MS | Agilent Model 7890A/5975 GC-MS Stationary phase: HP-PLOT/Q capillary column with particle trap, 30m×0.32 mm×20 µm. Carrier gas: He, flow rate: 1.5 mL min ⁻¹ Temperature program:50 °C for 1 min, increased to 200 °C at a rate of 10 °C min ⁻¹ ; held for 5 min; increased to 240 °C at a rate of 20 °C min ⁻¹ and held for 20min. | 43 | - | 0.2 ng g ⁻¹ | 0.5 ng g ⁻¹ |
| (Vázquez-Araújo et al., 2011) | furans | pomegranate juice | 7 | Headspace-SPME | Headspace GC-MS | Varian GC CP3800 coupled to Varian MS Saturn 2200 Stationary phase: VF-5MS column (30 m × 0.25 mm i.d., 1.0 µm film thickness). Carrier gas: He, flow rate: 1 mL min ⁻¹ Temperature program: 40°C held for 10 min, then increased 8°C min ⁻¹ to 180°C, and finally increased at 10°C min ⁻¹ to 280°C, where was held for 10 minutes. | 47.5 | ----- | ----- | ----- |
| (Varming et al., 2004) | furans | black currant juice | 3 | DHS | GC-MS | Hewlett-Packard G1800A S GC-MS system. Stationary phase: DB-Wax column (30 m 0.25 mm 0.25 µm). | 69 | 0.1-5 mg L ⁻¹ | ----- | ----- |

| | | | | | | | | | | |
|------------------------------|--------|---|----|--|----------------|--|-----|--|-------------------------------|-------------------------------|
| | | | | | | Temperature program: 40 °C for 10 min, increased with 6°C min ⁻¹ to 240 °C, and kept isothermal for 25 min. | | | | |
| (Furdiková et al., 2019) | furans | grape berries | 13 | SPME | GCxGC-HRTOF-MS | Pegasus GCxGCHRTOF-MS (Agilent 7890B GC) Stationary phase: DB-FFAP column (30m×0.25 µm×0.25 µm) and Rxi-17Sil column (1.6m×0.25mm×0.25 µm) Carrier gas: He, flow-rate: 1 ml min ⁻¹ Temperature program: 40 °C kept for 10 min, then increase by 2 °C.min ⁻¹ to final temperature 220 °C and kept for 5 min. | 120 | 0.13-3.25 mg L ⁻¹ | ----- | ----- |
| (Fernandes & Ferreira, 2000) | BAs | grape juice | 7 | ion-pair extraction and derivatization with heptafluorobutyric anhydride | GC-MS | A Hewlett-Packard 5890 GC coupled with Hewlett-Packard 5970B MS Carrier gas: He; Column:: A DB-5MS capillary column (30 m X 0.25mm ID. 0.25µm film thickness) Temperature program: 80°C hold for 1 min, increased at 15°C min ⁻¹ to 210°C, then increased at 20°C min ⁻¹ to 290°C and held constant at 290°C for 5 min. | 18 | 0.01-5 mg/L | <0.01 mg/L | <0.05 mg/L |
| (Cunha et al., 2011) | BAs | grape juice | 22 | LLE. And derivatization with isobutyl chloroformate | GC-MS | A 6890 Agilent GC coupled with a 5973N Agilent MS Carrier gas: He Column: HP-5MS capillary column (30 m × 0.25 mm i.d., 0.25 µm film thickness) Temperature program: 100 °C hold for 1.0 min, ramped to 160 at 10 °C min ⁻¹ , then ramped to 280 at 25 °C min ⁻¹ , and hold for 13.3 min. | 25 | 0.010-10 mg/L | < 0.001 mg/L | 0.01 mg/L |
| (Jastrzębska et al., 2015) | BAs | red currant, black currant, cherry juices | 5 | centrifugation, filtration, degassing | IC | A Metrohm IC 883 Basic IC plus with conductivity detector controlled by MagicIC Net Basic software . Stationary phase: Metrosept C Guard/4.0 quard column; Metrospet C 4-100/4.0 analytical column Eluent: 5 mM nitric acid; flow rate 0.5 mL min ⁻¹ | 40 | 0.5-5 mg L ⁻¹ 5-100 mg L ⁻¹ | 0.056-1.63 mg L ⁻¹ | 0.19- 3.27 mg L ⁻¹ |

898 BAs – biogenic amines; CSHLLE-DLLME - counter current salting-out homogenous liquid-liquid extraction combined to dispersive liquid-liquid micro-extraction; CSDF-ME - continuous sample
899 drop flow micro-extraction; DAD – diode array detector; DES – deep eutectic solvent; DHS – dynamic headspace; DLLME – dispersive liquid-liquid microextraction; ECD – electron capture detector;
900 FAAS – flame atomic absorption spectrometry; FID – flame ionization detector; FLD – fluorescence detector; GC- gas chromatography; GFAAS – graphite furnace atomic absorption spectrometry;
901 HPLC – high performance liquid chromatography; HRTOF-MS – high resolution time-of-flight mass spectrometry; IC - ion chromatography; ICP-OES – inductively coupled plasma optical emission
902 spectrometry; IL – ionic liquid; ILSFOD-LLME – ionic liquid-assisted liquid-liquid microextraction based on the solidification of floating organic droplets; IS-SULLE - in-syringe sugaring-out liquid-
903 liquid extraction; LC – liquid chromatography; LLE-liquid-liquid extraction; MS – mass spectrometry; MSDP – multiresidue matrix solid-phase dispersion; m-PFC - multi-plug filtration cleanup;
904 mSPE – magnetic solid phase extraction; NPD – nitrogen-phosphorus detector; QuEChERS - Quick Easy Cheap Effective Rugged Safe; PAHs – polyaromatic hydrocarbons; PCBs – polychlorinated
905 biphenyls; PTh IL-Mmt SPE - montmorillonite clay intercalated with ionic liquids co-deposited with polythiophene polymer coated electrochemically on solid-phase extraction; Va-d-µ-SPE – vortex-
906 assisted dispersive solid phase extraction; SPE – solid phase extraction; SPME – solid phase microextraction; ToFMS – Time-of-flight mass spectrometry; UV – ultraviolet/visible light detector

907

908 **5. Conclusions and future remarks**

909 From year to year, the demand for fruit juices increases. Particular interest can be observed for juices
910 produced from superfruits, which include berries. This is due to the fact that consumers pay more
911 and more attention to the composition of food products that they include in their daily diet. A very
912 important feature of food has become its health-promoting properties, and thus health benefits.

913 The presented literature review focuses on modern analytical methods that enable the determination of
914 analytes contained in fruit juices that may have health-promoting properties, but also those analytes
915 whose presence may be harmful to our health.

916 In the case of the determination of bioactive substances, spectrophotometric techniques are used
917 for preliminary studies to determine summary parameters, such as the total polyphenols content or total
918 anthocyanins content. In order to more accurately determine the composition of fruit juices,
919 chromatographic techniques, mainly liquid chromatography, are the most often used. These techniques
920 enable the determination of chemical compounds even at the trace level, and are also characterized by
921 good selectivity, accuracy and precision. The use of high-resolution chromatographic techniques enables
922 the detection of new potential active substances contained in fruit juices. However, analyzes often
923 require high consumption of organic solvents and complicated sample preparation procedures for the
924 isolation of analytes. In accordance with the principles of green organic chemistry, the aim is to replace
925 conventional solvents with greener ones, e.g. DES and solvents of biological origin. During the research,
926 the aim is also to miniaturize modern analytical methodologies while increasing the throughput, thus
927 enabling the determination of as many analytes as possible in a relatively short time. It should be noted
928 that in order to understand the nutritional potential and health-promoting properties of fruit juices, it is
929 necessary not only to determine bioactive substances, but also to study their metabolism in the human
930 body. Increasingly, both targeted and untargeted metabolic approaches are being used in research. When
931 establishing a chemical fingerprint and metabolic profiling, the key element is data analysis, during
932 which bioinformatics tools are used. Metabolomics makes it possible to find new bioactive compounds,
933 as well as new juice biomarkers that allow them to be distinguished.

934 In the case of contamination of juices with microbiological and chemical agents, it is very important
935 to find reliable methods to detect them. Due to the increase in the amount of possible food contamination
936 caused by industrialization and globalization, food safety assessment should be at the heart of the food
937 industry.

938 Finding fast, reliable and sensitive methods for detecting contaminants in fruit juices is essential for
939 assessing food quality and ensuring consumer safety. Spectroscopic techniques (AAS and ICP-EOS)
940 are mainly used for metal content analysis. For the determination of other pollutants (e.g. pesticides,
941 mycotoxins, phthalates, biogenic amines and others), chromatographic techniques (GC and LC)
942 are most often used. During the research, the aim is to develop modern analytical methodologies
943 enabling the determination of pollutants at lower and lower concentration levels. The ultimate goal of the
944 new methodologies should be selective and sensitive, miniaturized, automated and lab-independent
945 contamination determinations.

946 It should also be noted that metabolomics has potential as a screening tool for detecting adulteration of
947 juices, as well as their contamination. It can be a new strategy in the food industry, enabling quick
948 detection of any irregularities in the composition of fruit juices.

949 In the future, efforts should also be made to develop new analytical methods enabling the detection of
950 impurities and quality control during in-situ juice. Different types of sensors can be used for this purpose,
951 such as electronic noses, electronic tongues or electrochemical sensors.

952 In accordance with the principles of sustainable development, the industry strives to reduce the amount
953 of waste produced. Wastes obtained during the production of juices, such as pomace, seeds, skins, etc.,
954 still contain large amounts of bioactive compounds. Future research should therefore aim at developing
955 green methodologies for extracting bioactive compounds such as polyphenols, flavonoids or pectins
956 from fruit pomace.

957 In conclusion, comprehensive specifications for fruit juices should be established in the future. It is to be
958 hoped that modern analytical methods, as well as international cooperation between scientists, will
959 enable the development of such analytical tools that will guarantee that fruit juices entering the market
960 will be healthy and safe for consumers.

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