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Extraction of bioactives from fruit and vegetables: State of the art and perspectives

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► **To cite this version:**

Catherine M.G.C. Renard. Extraction of bioactives from fruit and vegetables: State of the art and perspectives. LWT - Food Science and Technology, 2018, 93, pp.390-395. 10.1016/j.lwt.2018.03.063 . hal-01925634

HAL Id: hal-01925634

<https://hal.science/hal-01925634v1>

Submitted on 16 Nov 2018

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Manuscript Number: LWT-D-17-02552R1

Title: Extraction of bioactives from Fruit and Vegetables: State of the Art and Perspectives

Article Type: Review article

Keywords: micronutrients, functional properties, stability, process intensification, solvent

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Abstract: Fruit and vegetables are rich in bioactive compounds that contribute to the prevention of a number of degenerative diseases. These components are also present, often in even higher concentrations, in the co-products from fruit and vegetable processing. Such fact, makes these co-products an attractive source for the extraction of bioactives, turning the extraction itself into an attractive valorization strategy for these co-products. There has been recently renewed interest in extraction methods, notably with a process intensification using physical phenomena and the search for alternative solvents.

This paper will present the main bioactives in fruit and vegetables, and their co-products, and the precautions to preserve these molecules in the food processing chain, with a main focus on the pros and cons of recently proposed extraction developments, particularly on extraction mechanisms, sample pre-treatment, and solvent choice.



UMR408 SQPOV

INRA
SCIENCE & IMPACT



Avignon, August 21, 2017

Dear Professor Sathe,

Please find attached the text of a short review which I would like to submit to LWT-Food Science and Technology, titled: "Bioactives in Fruit and Vegetables and their Extraction Processes State of the Art and Perspectives". The aim is a general overview of the newly proposed extraction methods and the specific challenges when they are used for extraction of bioactives from co-products of fruit and vegetables.

I am the sole author. The review has 3200 words, 2 tables and 49 references

Cordially

Catherine Renard

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Answer to reviewers

The manuscript is a short and focused review on an important topic. The author succinctly covers the important issues in this arena of research from a practical viewpoint and summarizes what is known and what should be looked at in the future. While discussing the issues the author explains the reasoning and possible causes/problems encountered in a lucid manner. Overall, the review is well written. A minor suggestion is to tidy up the write-up. A few points in this regard are noted below:

Thank you for your appreciation. I have had the manuscript corrected for English by a commercial service, which should have substantially tidied up the write-up, at least there were a number of English language corrections. (in blue)

1. Line 48, colour should be "color"?
2. Line 51, suggest use inexpensive instead of cheap
3. Line 80, sa(Brewer, 2011)ponins should be saponins (Brewer, 2011)
4. Line 85 (energy production---) should be (e.g., energy production, protein synthesis and others) to avoid the use of In a sentence.
5. Line 89, (anthocyanins? Is it meant to be- (anthocyanins).
6. Line 132, carotene should be carotene
7. Line 184, replace cheap with inexpensive or affordable

All of these have been done (in red)

8. Lines 310-319, Include a sentence or two either in this paragraph or any other place in the conclusion section about the lack of investigations addressing the in vivo absorption and bioavailability of the "functional" ingredients purported to be "bioactive". In addition there is a clear void of valid information based on in vivo studies that demonstrate, beyond reasonable doubts, efficacy of several reported "bioactive and/or functional ingredients". A good example is that of the time and again some reporting the functionality of "ginkgo biloba" where there is no proof it has, beyond reasonable doubt, efficacy in vivo in a living organism.

The author may consider adding a short table summarizing a list of "functional or bioactives" reported but not demonstrated to be effective in vivo (particularly in humans). The author has pointed this out in a subtle way when discussing the "chronic diseases and the role of extractives". May consider expanding that section with a couple of specific examples.

I totally agree with the reviewer; however this deserves a review (metaanalysis?) of its own and goes much beyond the scope of the present, more technical, review. I have therefore:

- Changed the title to make it clearer that my focus was on the extraction process itself
- -Added a number of qualifiers regarding the fact of "bioactivity" (in red)

Highlights

New extraction methods have received a lot of attention in the last 10 years

Most studied methods were microwave and ultrasound-assisted extraction

Alternative solvents are sought with improved eco-toxicity

More attention should be paid to pre- and post-extraction processes

Use of co-products is limited by their availability and limited choice of bioactives.

1 **Extraction of bioactives from Fruit and Vegetables: State of the Art and**

2 **Perspectives**

3

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17

18 **Abstract**

19 Fruit and vegetables are rich in bioactive compounds that contribute to the prevention of a
20 number of degenerative diseases. These components are also present, often in even higher
21 concentrations, in the co-products from fruit and vegetable processing. Such fact, makes
22 these co-products an attractive source for the extraction of bioactives, turning the extraction
23 itself into an attractive valorization strategy for these co-products. There has been recently
24 renewed interest in extraction methods, notably with a process intensification using physical
25 phenomena and the search for alternative solvents.

26 This paper will present the main bioactives in fruit and vegetables, and their co-products, and
27 the precautions to preserve these molecules in the food processing chain, with a main focus
28 on the pros and cons of recently proposed extraction developments, particularly on extraction
29 mechanisms, sample pre-treatment, and solvent choice.

30

31 Keywords: micronutrients, functional properties, stability, process intensification, solvent

32

33 **1. Introduction**

34

35 The increase in consumers' demand for naturalness and the trends towards plant-based
36 foods has sparked a renewed interest in fruit and vegetables and their co-products as
37 sources of bioactive and functional components. Most hydrocolloids (polysaccharides) as
38 well as a number of colorants (anthocyanins, carotenoids, betalains) or antioxidants
39 (rosemary leaf extract, E392) are extracted from plants. These have clear technological
40 functions in the foods and can be used to replace some synthetic additives. The concept of
41 "bioactives" is much more difficult to substantiate. A high level ("five-a-day") of consumption
42 of varied fruit and vegetables is associated with better health, and there has been great
43 interest in identifying the molecules behind this effect. In recent years, this has progressed
44 beyond the oversimplifying "antioxidant" hypothesis towards the identification of mechanisms
45 by which these molecules, or rather their metabolites, interact with specific targets in the

46 human organism (Dangles, 2012). However, there is still a lack of critical outlook on which
47 the actual activities of these “bioactives” are, and many papers use simplistic colorimetric
48 antioxidant assays as proxy for bioactivity.

49

50 Concurrently, there has been renewed interest in methods to obtain these bioactive or
51 functional components either for food uses, which are mostly relevant for molecules with
52 functional properties (texture, color, antioxidant, aroma or taste), as food supplements,
53 though their use as isolated molecules has proven disappointing, or as cosmetics. For
54 economic reasons, the preferred sources are the co-products from fruit and vegetable
55 processing as these are inexpensive and often highly concentrated in some bioactives. The
56 ideal situation would be a complete use of the fruit and vegetable biomass in a biorefinery
57 that would take into account food uses, high value bioactives, and the use of the remaining
58 bulk material. Major issues for development of such a biorefinery lie in conciliating the
59 different aims and qualities of the products as well as in dealing with availability issues.

60

61 Classical extraction techniques such as maceration or supercritical fluid have been
62 supplemented with many “assisted” extraction techniques where an additional physical
63 phenomenon is used to intensify the process. There has been an explosive growth of
64 publications on these topics since the year 2000 (Kala, Mehta, Sen, Tandey, & Mandal,
65 2016; Mandal & Tandey, 2016). The physical phenomena range from ultrahigh pressure to
66 void (instant controlled pressure drop), from microwave to ultrasound or electric fields. Many
67 of these techniques were first developed for analytical purposes in order to obtain a true
68 quantification of the bioactive molecules, and some have proven to be profitably up-scalable,
69 at least at pilot-scale level. Newer challenges concern the solvents used in the extraction, as
70 REACH (Registration, Evaluation, Authorisation and Restriction of Chemicals regulation)
71 comes into application and costs of solvent recovery become more and more significant.
72 Biosourced solvents, subcritical liquids or ionic liquids thus appear now more frequently in
73 the scientific literature and also pose specific challenges.

74

75 **2. Bioactives in fruit and vegetables: chemical classes and activities**

76

77 The very notion of “bioactive” or bioactive compounds can have different definitions in
78 scientific literature. It is generally associated with the existence of a positive effect on chronic
79 pathologies, with different levels of proof (Amiot, Coxam, & Strigler, 2012). In the most
80 common meaning of the term, it does not cover vitamins or dietary fibers, although they are
81 proven to be indispensable for human health, or compounds with demonstrated acute
82 pharmacological or negative activities (notably alkaloids). Indeed, fruit and vegetables by
83 definition have no detectable acute effect in a normal dietary pattern, at least in the Western
84 world, while many drugs were originally identified in plants, giving rise to the large field of
85 phytochemistry. Some chemical classes such as saponins (Brewer, 2011) are “borderline”.
86 The level of proof or activity needed for a health claim appropriate to a functional food or
87 nutraceutical is therefore a tight balance between the demonstration of a pharmacological
88 effect and unsubstantiated claims. The best evidence of such fact is that very few molecules
89 or extracts have been able to pass the stringent evaluation of claims made by EFSA
90 (European Food Safety Agency).

91

92 In terms of plant physiology, most of these compounds are secondary metabolites, i.e. they
93 are not necessary to the plants’ basic metabolism (e.g. energy production, protein synthesis,
94 and others). It is generally believed that their role *in planta* concerns interactions with the
95 outer environment, where they may act as protective agents against UV radiation (flavonols),
96 as deterrents against herbivores (glucosinolates, some polyphenols), or as attractants for
97 pollination (anthocyanins), among other properties and functions. The underlying chemical
98 complexity explains that relatively few of these molecules have been studied for their action
99 in preventing life-style diseases or the relatively low levels of proof for many of them. This
100 may also explain why most developments have been focused on a few classes that are

101 either remarkably abundant (like the polyphenols or carotenoids) or remarkably active
102 (glucosinolates, isoflavones).

103

104 Table 1 summarizes the classes of compounds that are most commonly called “bioactives”
105 and which can be found in fruits or vegetables *sensu largo*, i.e. including herbs and spices,
106 but not medicinal plants. Some of the classes are very specific of a given botanical family, for
107 example the capsaicinoids (Capsicum) or glucosinolates (Brassica) while others are very
108 widespread, like the phenolic acids or flavonols.

109

110 Many of these compounds have antioxidant properties. It is now generally recognized that
111 the health effects of bioactives are mediated by specific interactions of their circulating
112 metabolites with cellular targets (Dangles, 2012) and not antioxidant activity per se. However
113 the antioxidant properties may be relevant when focussing on food or cosmetics [stabilization](#),
114 for [the](#) replacement of synthetic antioxidants such as butylhydroxytoluene or [the](#) prevention
115 of oxidation of polyunsaturated fatty acids in the gut (Brewer, 2011).

116

117

118 **3. Co-products from fruit [and](#) vegetable processing and their stabilization**

119

120 Many of the secondary metabolites are concentrated in the outer, least palatable parts of fruit
121 and vegetables and [are](#) therefore present in high concentrations in [the](#) co-products
122 processing (Baiano & Del Nobile, 2016; Banerjee et al., 2017; Lavelli & Torresani, 2011; Le
123 Bourvellec et al., 2011; Martins & Ferreira, 2017; Strati & Oreopoulou, 2014). However, these
124 high concentrations are relative: polyphenols may reach from 10 to 50 g/kg [DW \(dry weight\)](#)
125 in pomaces from fruit juice extraction (Juśkiewicz et al., 2015; Kolodziejczyk et al., 2009;
126 Pieszka, Gogol, & Pietras, 2015), while carotenoids [yield under 10 g/kg DW](#) even in tomato
127 by-products (Kalogeropoulos, Chiou, Pyriochou, Peristeraki, & Karathanos, 2012; Lavelli &

128 Torresani, 2011), and dietary fibers represent over 500 g/kg DW (Garcia, Valles, & Lobo,
129 2009; Kolodziejczyk, et al., 2009).

130

131 A major limiting factor is that of an eventual co-product resource as the question of bioactives
132 cannot be addressed from the target property but from availability. We do not ask ourselves
133 “what can I extract this molecule of interest from” but “what can I do with the co-product I
134 have”. Another point to be considered is the high variability of the resource: concentrations of
135 the target compounds are likely to vary, sometimes over 10-fold, depending on the exact
136 variety (and its maturity stage) (Garcia et al., 2009).

137 The alternative is the establishment of a specific production chain, which demands a
138 demonstrated and sufficient final value of the bioactive. Indeed, there are often alternatives
139 using specific plants or other parts of plants, which are economically more viable than relying
140 on fruit and vegetables. For example, lutein is extracted from *Tagetes* flowers and alfalfa; β -
141 carotene from specific varieties of carrot with high concentrations; phloridzin is much more
142 abundant in apple leaves than in apple fruit (Gaucher et al., 2013).

143

144 In practical terms, this means that research on bioactives from fruit and vegetables is still
145 very much “opportunity-driven”, i.e. focused on products that meet conditions of availability
146 and potential interest. The availability of a co-product is in turn linked to the existence of a
147 production chain. Thus, the most studied sources (Galanakis, 2012) are pomace from grapes
148 (wine) (Barba, Zhu, Koubaa, Sant'Ana, & Orlie, 2016), tomato (pulp and concentrate),
149 apples (juice), berries (juice), peels from Citrus (juice) or mango (puree or dried mangoes),
150 sugar-beet pulp (sugar refining), waste water from olive processing (olive oil), and peels from
151 onion or salad (fresh-cut onions or salads). Dietary fibers and polyphenols are the main
152 chemical classes of interest, only with tomato co-products being abundant and rich in
153 carotenoids.

154

155 The treatments that take place for the initial product preparation (for example preparation of
156 cloudy or clear juice, Kolodziejczyk et al., 2009) as well as those used for the stabilization of
157 the co-product will also impact its composition and potential as a source of bioactives. Two
158 phenomena must be taken into account when envisioning the extraction of polyphenols from
159 e.g. apple pomace: one is that the molecules present in the pomace may not be those of the
160 original fruit, as there can be extensive enzymic oxidation during the juice extraction and
161 pomace processing (Heras-Ramirez et al., 2012) as well as chemical degradation; and the
162 other is that some polyphenols, notably the condensed tannins, form strong adducts with the
163 cell wall material (Renard, Watrelot, & Le Bourvellec, 2017) and may become un-extractable.
164 Whether the pomace is initially treated to inhibit enzymes or not, the drying method and
165 temperature as well as its final water content and storage temperature will all impact the final
166 composition (Heras-Ramirez, et al., 2012; Lavelli & Corti, 2011; Lavelli & Torresani, 2011;
167 Yan & Kerr, 2013). Carotenoids, in contrast to polyphenols, appear less stable at the lower
168 a_w values (Lavelli & Torresani, 2011).

169

170 One major difficulty in going from a laboratory demonstration to actual industrial production
171 lies in the seasonal availability of fruit and vegetables and also of their co-products. For
172 example, industrial tomatoes are harvested and processed over a period of about two
173 months. Therefore, their pomace, although remarkably high in lycopene, is only available
174 fresh during this short period. The implementation of co-products, which are prone to
175 microbial waste, may require immediate processing or a stabilization step, most often by
176 drying, which may lead to some loss of bioactives by thermal degradation of the more fragile
177 molecules or interfere with the extraction procedure itself (Rajha et al., 2014).

178

179

180 **4. Recent developments in extraction methods**

181

182 4.1. Conventional methods

183 The **most** classical methods for extraction of bioactives rely on maceration (with more or less
184 intense stirring) in a solvent of appropriate polarity (Galanakis, 2012). A first improvement is
185 increasing the extraction temperature for better dissolution and lower solvent viscosity, as
186 done e.g. in heated reflux extraction (which also takes advantage of concentration equilibria)
187 or in a Soxhlet apparatus (which has the **further** advantage of separating the soluble from the
188 insoluble fractions). Decreasing particle size is also a factor that has long been taken into
189 consideration as it facilitates mass transfer and thus increases extraction speed (and yields).
190 Supercritical fluids, and among them mostly carbon dioxide, have been used industrially in
191 the food and cosmetics industries. Supercritical carbon dioxide has a low critical temperature
192 and pressure (31 °C, 7.39 MPa), which **is** of interest for labile molecules, and its polarity can
193 be increased by adding co-solvents (e.g. ethanol). It is GRAS (**Generally Recognized as**
194 **Safe**) and non-explosive, relatively **affordable**, and the extracted compounds can be
195 recovered by evaporation of the gas. It is commonly used for **the** extraction of essential oils
196 and has a good potential for carotenoids.

197 Enzymic pre-treatments **have** also been used in improving extraction yields, because it has
198 long been **acknowledged** that plant cell walls are one of the limiting factors. However pre-
199 treatments with cellulases or pectinases demand long incubations (typically a few hours) in
200 aqueous media.

201

202 4.2. Emerging technologies

203

204 More recent developments concern the use of non-thermal concepts to facilitate **the**
205 extraction without risking **the matrix** overheating **while** decreasing energy use. Recent
206 reviews describe **the** application of conventional and emerging technologies to the extraction
207 of various classes of bioactives (Ameer, Shahbaz, & Kwon, 2017; Gil-Chavez et al., 2013;
208 Lu, Ho, & Huang, 2017; Poojary et al., 2016; Wijngaard, Hossain, Rai, & Brunton, 2012). All
209 aim to destroy cell integrity by elimination of the cell membranes or cell walls. The most

210 studied applications since 2005, as identified from scientific publication trends, concern
211 microwave and ultrasound-assisted extraction (Mandal & Tandey, 2016).

212

213 Microwaves are electromagnetic waves, generally used at 2.45 GHz, which interact with
214 polar molecules (typically water, ethanol...) and generate heat (Zhang, Yang, & Wang,
215 2011). In microwave-assisted extraction, the moisture inside the cell is heated and its
216 evaporation increases the porosity of the biological matrix, which in turn allows **the** better
217 penetration of a solvent (Ho, Ferruzzi, Liceaga, & San Martín-González, 2015). The elevated
218 temperature also generally increases solubility and improves yield. Its main advantage is **a**
219 reduction of the extraction time and solvent use. Elevated temperatures may still result in
220 some degradation of the more labile molecules (Cardoso-Ugarte, Sosa-Morales, Ballard,
221 Liceaga, & San Martín-González, 2014).

222

223 Ultrasound (> 20 KHz) improves extraction through acoustic cavitation: above certain energy
224 levels, the acoustic waves interact with the solvent and dissolved gas by creating free
225 bubbles that can **expand** to a maximum size and violently collapse, generating locally
226 extreme heat and pressures (Tiwari, 2015). Due to this cavitation phenomenon, the cell walls
227 can be ruptured, providing channels for solvent access, and mass transfer is improved
228 (Pananun, Montalbo-Lomboy, Noomhorm, Grewell, & Lamsal, 2012). The small size of the
229 bubbles means that the heat generated upon collapse can be dissipated rapidly and **the** bulk
230 temperature increase can **remain** limited (Viroto, Tomao, Le Bourvellec, Renard, & Chemat,
231 2010). However, liquid-solid separation may be hindered **since** ultrasound can lead to the
232 swelling and disintegration of the plant material (Viroto et al., 2010).

233

234 Although many articles report the “optimization” of microwave or ultrasound treatments for
235 extraction, many of them fail in their purpose. This is particularly true **for the** frequent use of
236 response surface methodology (**RSM**) to conclude that **the** highest yields are obtained at **the**
237 highest power, longest durations, highest temperature and highest solvent/solid ratio. All can

238 be expected and [this does not correspond to the](#) correct use of RSM. If the optimum is at an
239 extreme of the experimental plan, by definition the limits of the RSM were not well chosen, or
240 the authors have used statistics to avoid thinking about the actual mechanisms.

241

242 Among electrotechnologies, different pulse protocols and intensities of electric fields have
243 been used to generate cell disintegration and thus enhance extraction of intracellular
244 compounds. The most studied is Pulsed Electric Fields ([PEF](#)), in which the sample is
245 submitted to very short periods ([from](#) several nanoseconds to several milliseconds) of an
246 intense electric field. This causes the formation of pores in the cell membranes
247 (electroporation), which helps in solvent diffusion and facilitates mass transfer (López,
248 Puértolas, Condón, Raso, & Ignacio, 2009; Puértolas, Cregenzán, Luengo, Álvarez, & Raso,
249 2013; Rastogi, 2003). PEF is also used to increase juice yields, i.e. non selective extraction
250 of intracellular fluids (López, Puértolas, Condón, Raso, & Ignacio, 2009)

251

252 Pressurized liquid extraction (or accelerated solvent extraction) relies on increased
253 temperature and pressure in a solvent kept below its boiling point to enhance mass transfer
254 and modify surface equilibria in [the](#) solid – liquid extraction (Mustafa & Turner, 2011).

255 Pressure is [primarily](#) used to maintain the solvent in the liquid phase, though it may also
256 facilitate solvent entrance in the pores of the matrix. It is commonly used in [the](#) analytical
257 quantification of bioactives [and](#) also pesticides. The elevated temperature also modulates the
258 polarity of the solvent and thus its extraction selectivity.

259

260 Negative pressure cavitation is a patented technology that aims to produce cavitation by
261 depression to generate an intense erosion of solid particles and increase turbulence and
262 mass transfer from the solid matrix to the solvent (Roohinejad et al., 2016).

263

264 Another potential pre-treatment is high pressure homogenization (Corrales, Garcia, Butz, &
265 Tauscher, 2009; Xi, 2017), a wet milling process where plant particles are disintegrated by

266 high intensity mechanical stresses as a consequence of the liquid flow through a
267 homogenization chamber at high pressures (50-500 MPa). It can be used on wet samples.

268
269 Another method which has been proposed for sample disintegration prior to extraction is
270 [instant controlled pressure drop \(DIC for Détente Instantanée Contrôlée\)](#) (Allaf et al., 2013).

271 In DIC, the sample is subjected to saturated steam [for a short time](#) and then to a sudden
272 pressure [drop](#) at low pressures. This causes [the](#) instantaneous vaporization of water,
273 resulting in [a](#) cell wall expansion and rapid cooling. The instantaneous vaporization allows
274 [the](#) recovery of essential oils while polyphenols or carotenoids are more easily extracted from
275 the remaining fine powder (Allaf et al., 2013). Similar results may be obtained by intense
276 grinding, provided [that](#) care is taken to prevent temperature increase during grinding.

277

278 4.3. Alternative solvents

279

280 The solvent choice is a concern as it impacts selectivity, [the](#) removal and disposal method,
281 costs and safety. The REACH directive has notably been an incentive for “green” solvents in
282 replacement [of](#) *n*-hexane. For example, among alcohols, ethanol is often proposed due to its
283 low boiling point and GRAS status but may not be efficient for less polar molecules such as
284 carotenoids. Water is also often tested, alone or in mixture with ethanol or acetone, in spite
285 of its high energy requirements for evaporation (Wiboonsirikul, Hata, Tsuno, Kimura, &
286 Adachi, 2007). More apolar solvents, mostly terpenes (limonene, alpha-pinene), have also
287 been proposed (Filly, Fabiano-Tixier, Fernandez, & Chemat, 2015). Another option is the use
288 of subcritical solvents such as liquefied gases (e.g. *n*-butane), which may ally low polarity
289 and facile solvent elimination by decompression at or close to room temperature (Rapinel et
290 al., 2017). However, safety issues must be addressed for these new solvents.

291

292 Recently, ionic liquids have been proposed for [the](#) extraction of bioactives. Ionic liquids
293 *sensu strictu* are organic salts in a liquid state. Although they are viscous, their polarity can

294 be adjusted across a wide range of hydrophobicity/hydrophilicity, and some of them are
295 distillable in conditions compatible with the recovery of bioactives (Almeida et al., 2014; Lu et
296 al., 2013). The most recent trend in this field is the use of “natural deep eutectic solvents”
297 (NADES), which are combinations of natural components such as sugars, organic acids or
298 aminoacids with tailored solvent properties (Radosevic et al., 2016). However, solvent
299 removal becomes more complex.

300

301

302 **Conclusion**

303

304 A lot of experimental work has been devoted in the last 10 – 20 years to the development of
305 “alternative” technologies for the extraction of bioactives from plants, including fruit and
306 vegetables. It is time for this body of experimental data, which mostly relies on the statistical
307 optimization of yields, to give place to more mechanistic oriented research. In addition, post-
308 extraction treatments (elimination of the solvent, purification...) are the poor relatives in this
309 research.

310

311 A number of technical issues have to be addressed before and after extraction: will the raw
312 material be stabilized or only extracted fresh, i.e. over a short period? If it is stabilized, how
313 shall it be done with the least loss of bioactive? Is a purification step needed? Most of these
314 molecules are only present in low concentrations. Therefore, their extraction will still leave
315 large amounts of waste: can that waste in turn become a co-product? This requires
316 anticipation and conceiving the whole chain in an integrated manner for the valorization of
317 the whole biomass.

318

319 Two main conditions must be met for the extraction of bioactives from fruit and vegetables to
320 be economically interesting:

321 - The activity or functionality of the molecules must have a market potential;

322 - It must be present in an amount **that is** compatible with its market in an **inexpensive**
323 co-product – or the market potential must be sufficient for specific production.

324
325 Color **and** antioxidant capacity are properties relatively easy to assess, which may explain
326 why they constitute the main fields of application for extracts from fruit and vegetables.
327 Having sufficient data for a health claim or nutraceutical use is a totally different proposition
328 **since** the cost of demonstrating the claim becomes high. **This may explain the lack of valid**
329 **information based on *in vivo* studies that demonstrate, beyond reasonable doubts, the**
330 **efficacy of several reported "bioactive" ingredients.**

331
332 All of this may explain why, in spite of **the wide** research, few fruit and vegetable co-products
333 are actually used at industrial scale for **the** production of bioactives (Galanakis, 2012):
334 anthocyanins from grape skin, (oenological) tannins from grape seeds, lycopene from tomato
335 waste (Strati & Oreopoulou, 2014), polyphenols from olive mill waste water and flavonoids
336 from citrus peel. **The** production of oil from grape seeds or kernels of plums, apricots and
337 peaches, dietary fibers from vegetable wastes and pectin extraction (from citrus peel, apple
338 pomace or sugar-beet pulp) may also be counted in this valorization of fruit and vegetable
339 co-products. The few successful examples need to be carefully analyzed, not from a
340 technological **perspective** but from a socio-economic and legislative point of view to identify
341 the factors of their success.

342

343 **Acknowledgements**

344 This article summarizes an oral presentation done at Bio2Actives 2017, Quimper, France.
345 The author thanks the organizing staff and **especially** S. Guillotin for their encouragement.

346

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348

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- 1 Table 1: The main chemical classes of bioactive compounds in fruit and vegetables (from
 2 Renard et al., 2012)

Chemical classes	Main Fruit and vegetable sources	Properties of interest
Terpenoids		
Monoterpenes	“herbs”, citrus	Aroma, digestion, antiseptic...
Diterpenes : carnosic acid	“herbs” (rosemary)	Aroma, antioxidant
Triterpenes: Phytosterols,	Plant oils	Cardiovascular health
Saponins	Soy, chestnut	
Tetraterpenes: Carotenoids:		
Carotenes: β-carotene	Orange fruit and vegetables, carrot	Color, provitamin A
lycopene	Tomato, watermelon	Color, prostate cancer
Xanthophylls: lutein	Green vegetables	Age-related macular degeneration
Phenolic compounds		
Flavonoids		
Flavonols	Onions, fruit, vegetables	Antioxidant
Anthocyanins	Red fruits, berries	Color, antioxidant
Flavanols and proanthocyanidins	Tea, wine, chocolate, cider, fruits	Astringent, Antioxidant, enzyme inhibition
Flavanones	Citrus	Antioxidant
Isoflavones	Soy, legumes	Phyto-œstrogen
Non flavonoids		
Phenolic acids	Coffee, fruits and vegetables	Antioxidant
Lignans	cereals	Phyto-estrogen
Hydrolysable tannins	Strawberry, fruits	Astringent, antioxidant
Stilbens: resveratrol	Wine, peanut (stressed)	Heart disease?
Tyrosol and derivatives	Olive	Antioxidant
Coumarins:	Apiaceae	Photosensibilisation
Sulphur-containing compounds		
Thiosulfinates	Allium	Aroma, antiseptic
Glucosinolates	Brassicaceae	Aroma / burning taste, thyroid hypertrophy, inhibition of <i>Helicobacter pilori</i> ...
Nitrogen-containing compounds		
Capsaicinoides	Capsicum	Burning taste, analgesic
Betalains	Caryophyllaceae	Color

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6 Table 2: Conventional and emerging technologies for the extraction of plant bioactives

Method	Advantage	Disadvantage
Extraction methods		
Maceration	Low investment cost; modulation of selectivity by solvent choice	Long; low recovery
Heating reflux, Soxhlet	Low investment cost; increased yields	High temperature; solvent
Supercritical fluid extraction	Low temperature; high yields; mostly for molecules of low polarity but can be modulated	High investment costs
Microwave-assisted extraction	Reduction of processing time and solvent use	Locally high temperatures; polar solvents.
Ultrasound-assisted extraction	Reduction of processing time, low temperature	Swelling of the plant material
Pressurized solvent extraction	Reduction of processing time and solvent use	Investment costs; temperature; low throughput
Pulsed electric fields	Reduction of processing time and solvent use	Requires conductivity; activity of enzymes
Tissue destructuration methods		
Extensive grinding	Facilitated mass transfer, can be carried out on dry samples	Risk of heating during grinding
Enzyme-assisted extraction	Facilitated extraction from a plant tissue	Additional long operation in wet conditions
Negative pressure cavitation	Moderate temperature, possibility of anaerobic conditions	Lack of background data
High pressure homogenization	Increased yields by tissue disintegration	No selectivity, requires separation
Instant controlled pressure drop	Increased yields by tissue disintegration	Pre-treatment only; requires pre-drying.

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Supplementary Material

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