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Review

Updating the research on prodelphinidins from dietary sources

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ABSTRACT

Polyphenols are common constituents of foods of plant origin with thousands of different compounds comprising a wide variety of molecules. Prodelphinidins (PDs) belong to the proanthocyanidin (PA) or condensed tannins sub-group. Not so studied like their similar procyanidins (PCs), PDs have attracted attention over the last years, mostly due to their antioxidant capabilities in biological systems.

This review is focused in all PDs detected so far in dietary sources, highlighting the principal features of the detection, synthesis and possible health benefits.

Recently, new methodologies for the analysis of dimeric PDs have been achieved through isolation, purification and high-performance liquid chromatography mass spectrometry analysis. Concerning oligomeric and polymeric PDs, spectrometric studies are limited probably owing to their coexistence with a large number of other PA molecules, their low concentration and the high number of isomers.

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Contents

1. Introduction

Proanthocyanidins (PAs) or condensed tannins are one of the most important polyphenolic compounds in plant-derived food like fruits, cereals, and beverages. They are secondary metabolites synthesized by plants during normal development and in response to stress conditions such as fungal and bacterial infections (Skipp, 1977), UV radiation (Koes, Quattrocchio, & Mol, 1994), water stress, among others. Their

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Abbreviations: PA, proanthocyanidins; PC, procyanidin; PD, prodelphinidin; C, catechin; EC, epicatechin; (E)C, epicatechin or catechin; GC, gallocatechin; EGC, epigallocatechin; (E)GC, epigallocatechin or gallocatechin; GCG, gallocatechin gallate; ECG, epicatechin gallate.

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characteristic structure is formed by two phenyl rings A and B, and one heterocyclic ring C (Fig. 1). The consecutive units of condensed tannins are linked through an interflavonoid bond between C4 and C8 or C6 (Hemingway, 1989). Based on the hydroxylation pattern of rings A and B, PAs can be divided into procyanidins (PCs), propelargonidins and prodelphinidins (PDs).

PDs are not as studied as PCs. They seem to be less abundant in dietary sources and did not attract researchers' attention until recently. In fact, the only research done on PDs until the late 90s was related to the brewing industry due to their high content on barley and hop. These compounds (PAs and PDs) are important for that industry because they contribute to the bitter and astringent character of beer and its oxidative stability. The lack of commercial standards, proper detection and identification analytical methods, and also the lack of synthesis pathways did not help.

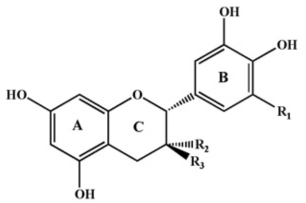
The different classes of PAs (monomers, PCs and PDs) became research material of interest in the last years especially because of their taste properties (Baxter, Lilley, Haslam, & Williamson, 1997; Soares, Brandão, Mateus, & De Freitas, 2015) and perceived health and biological properties. Indeed, it was described that these compounds could prevent cardiovascular diseases and some types of cancer (Chen, Liu, & Zheng, 2014; Fujii, Toda, Matsumoto, et al., 2013; Santos-Buelga & Scalbert, 2000); possess antioxidant and free radical scavenging activities (Plumb, De Pascual-Teresa, Santos-Buelga, Cheynier, & Williamson, 1998; Saint-Cricq de Gaulejac, Provost, & Vivas, 1999; Teissedre, Frankel, Waterhouse, Peleg, & German, 1996; Zhou et al., 2014); inhibition of tumor initiation and promotion in skin and other organs (Gali, Perchellet, Gao, Karchesy, & Perchellet, 1994; Meeran, Vaid, Punathil, & Katiyar, 2009; Valcic et al., 1996); antibacterial and angioprotective properties (Taguri, Tanaka, & Kouno, 2004; Vennat et al., 1988); and inhibition of platelet aggregation (Chang & Hsu, 1989; Morel et al., 2014).

2. Prodelphinidins identification and analysis

The quantification of phenolic compounds in plant materials is influenced by their chemical nature, the extraction method employed, sample particle size, storage time and conditions and presence of interfering substances such as waxes, fats, terpenes and chlorophylls.

Because of the large number of polyphenols in natural sources and the low levels of some of them, their analysis requires preliminary fractionation, isolation and purification steps prior to analytical analysis such as high-performance liquid chromatography (HPLC). Standard procedures include enrichment techniques such as solid-phase extraction, liquid–liquid extraction and column chromatography fractionation. Although these methods generally allow a good, but obviously not complete purification of samples, they are time-consuming and could also result in the loss of compounds.

Since the 1980s that different chromatographic procedures have been employed for tannin identification (Herderich & Smith, 2005) and quantification by using UV–Vis and/or fluorescence detectors (Naczk & Shahidi, 2004), depolymerization by interflavan bond cleavage by thiolysis (Czochanska, Foo, Newman, & Porter, 1980; Rigaud, Perez-Ilzarbe, Da Silva, & Cheynier, 1991) or phloroglucinolysis (Kennedy, Ferrier, Harbertson, & des Gachons, 2006; Kennedy & Jones, 2001). The more common nucleophiles for acid-catalyzed cleavage include benzylhydrosulfide (*syn*. phenylmethanethiol, toluene-a-thiol), and 1,3,5-trihydroxybenzene (*syn*. phloroglucinol) being the methods referred to as thiolysis and phloroglucinolysis respectively (Foo & Porter, 1978; Thompson, Jacques, Haslam, & Tanner, 1972). These methods can give information about the sample degree of polymerization and galloylation. However, thiolysis may result in inconsistencies attributed to overestimation of the degree of polymerization due to epimerization and disproportionation of its products (Porter, 1988). Besides that, A-type linkages are known to resist thiolytic degradation (Gu et al., 2003), resulting in the release of A-type dimeric terminal units (not monomers) and A-type benzylthioethers extension units.



R_1 = H; R_2 = H; R_3 = OH: Catechin (C)
R_1 = OH; R_2 = H; R_3 = OH: Gallocatechin (GC)
R_1 = H; R_2 = OH; R_3 = H: Epicatechin (EC)
R_1 = OH; R_2 = OH; R_3 = H: Epigallocatechin (EGC)
R_1 = OH; R_2 = OGallate; R_3 = H: Epigallocatechin gallate (EGCG)

Fig. 1. Chemical structures of PA monomers.

As PDs (like general PAs) normally co-exist as different isomers, different proportions of these compounds, for example in wine, may result in desirable qualities or defaults (Chira, Pacella, Jourdes, & Teissedre, 2011). Thus, obtaining more information about the exact tannin composition of food and/or beverages is recommended other than the total phenolic content and the mean degree of polymerization and galloylation. Such an approach involves PD structural knowledge (the use of synthetic PDs may be necessary) and standard HPLC-UV and HPLC-MS chromatograms for comparison.

Another important issue in the investigation of PDs arises from the abbreviated nomenclature used to identify and distinguish PCs from PDs. Considering that a PC can be described as a compound that releases cyanidin in acid medium when heated (Bate-Smith, 1954) and a PD as a compound that releases delphinidin in the same conditions, then for instance, a C-EGC dimer cannot be named a PD. A suggestion for PC and PD identification nomenclature taking into account the already adopted one is reported elsewhere (Teixeira, Azevedo, Mateus, & de Freitas, 2016) and described in Table 1.

2.1. Extraction and fractionation

Each type of plant material contains different quantities and qualities of phenolic acids, anthocyanins, PAs, flavones, etc. Usually the plant materials are crushed or grounded and macerated in a solvent or solvent system. The polarity of the solvent(s) used also affects the phenolic compounds solubility. Methanol, ethanol, acetone, water, ethyl acetate and also propanol and dimethylformamide are frequently used for the extraction of phenolic compounds (Antolovich, Prenzler, Robards, & Ryan, 2000). Some studies also refer that richer phenolic extracts can also be obtained upon heating the extracting mixture (Khanal, Howard, & Prior, 2010; Pacheco-Palencia, Mertens-Talcott, &

Table 1
Suggested identification nomenclature for PCs and PDs. Reprinted with permission from (Teixeira et al., 2016).

Compound	PC	PD
B1	EC-(4β-8)-C	EGC-(4β-8)-C
B2	EC-(4β-8)-EC	EGC-(4β-8)-EC
B3	C-(4α-8)-C	GC-(4α-8)-C
B4	C-(4α-8)-EC	GC-(4α-8)-EC
B5	EC-(4β-6)-EC	EGC-(4β-6)-EC
B6	C-(4α-6)-C	GC-(4α-6)-C
B7	EC-(4β-6)-C	EGC-(4β-6)-C
B8	C-(4α-6)-EC	GC-(4α-6)-EC
B9	EC-(4β-8)-GC	EGC-(4β-8)-GC
B10	EC-(4β-8)-EGC	EGC-(4β-8)-EGC
B11	C-(4α-8)-GC	GC-(4α-8)-GC
B12	C-(4α-8)-EGC	GC-(4α-8)-EGC
B13	EC-(4β-6)-EGC	EGC-(4β-6)-EGC
B14	C-(4α-6)-GC	GC-(4α-6)-GC
B15	EC-(4β-6)-GC	EGC-(4β-6)-GC
B16	C-(4α-6)-EGC	GC-(4α-6)-EGC

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