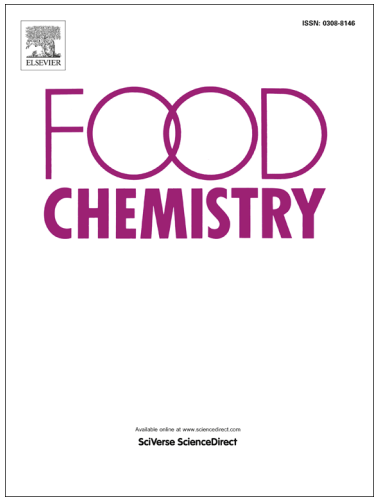

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New insight into the unresolved HPLC broad peak of Cabernet Sauvignon grape seed polymeric tannins by combining CPC and Q-ToF approaches

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Abstract

Polymeric tannins from grapes have always been reported as an unresolved broad peak in HPLC chromatograms, and this has severely limited their identification to date. This study aimed to disassemble this broad peak and explore the polymeric tannin molecules inside. By applying centrifugal partition chromatography (CPC), an efficient separation approach was developed to split the broad peak of grape seed tannins into fractions. Then, the fractions were analyzed by Q-ToF (quadrupole time-of-flight mass spectrometry) to determine the corresponding structures of the tannins. The results suggest that grape seed polymeric tannins were eluted consecutively according to their degree of polymerization (DP). Condensed tannins identified in wine grape seed have a range of DP and degree of galloylation (DG) up to 20 and 11, respectively. The molecular mass of the largest molecule detected was 6067. To our knowledge, this is the first report to offer an insight into the broad peak of polymeric tannins found with HPLC and to characterize the tannins with a DP up to 20 as shown by HRMS and MS/MS data.

Keywords: condensed tannins; grape; CPC; Q-ToF; polymer

1. Introduction

Condensed tannins are oligomeric and polymeric forms of flavan-3-ol monomer units that are also known as proanthocyanidins in the domain of phytochemistry. Owing to their considerable contribution to wine sensory perception (W. Ma, Guo, Zhang, Wang, Liu, & Li, 2014) and other bioactivities (Rasines-Perea & Teissedre, 2017), the condensed tannins derived from grape and wine have attracted the attention of wine chemists for decades. As the unique source of condensed tannins in wine (J.A. Kennedy, Saucier, & Glories, 2006), grape is easier to study than wine since grape tannins occur in their initial forms prior to any polymerization or oxidation reactions during vinification or wine aging. Until now, numerous studies have investigated the oligomeric condensed tannins in grape (Ge, Zhu, Kazuma, Wei, Yoshimatsu, & Komatsu, 2016; Lin, Sun, Chen, Monagas, & Harnly, 2014; Wen Ma, Waffo-Teguo, Jourdes, Li, & Teissedre, 2016). While polymeric tannins are much more abundant in grape than oligomers (K. Chira, Schmauch, Saucier, Fabre, & Teissedre, 2009), little is known about their chemical structure or composition. The characterization of grape