

Chromatographic evaluation of the components of grape skin extract used as food additives

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Abstract

Grape skin extract, a food manufacturing agent registered in the List of Existing Food Additives in Japan, was evaluated by high performance liquid chromatographic (HPLC) method. Chemical constituents of these extracts were separated by repeated column chromatography, and 12 compounds were isolated and characterized as tryptamine, syringic acid, vanillic acid, ethyl gallate, (+)-catechin, (-)-epicatechin, luteoliflavan, quercetin, quercetin 3-*O*-glucuronide, myricetin 3-*O*-glucoside, procyanidin B-1, and procyanidin B-2 by spectroscopic methods. The presence of malvidin 3-*O*-glucoside as the major anthocyanin was confirmed by HPLC. A broad peak forming a swollen base line was attributed to a B type of proanthocyanidins, *i.e.*, a condensed tannin oligomer, the number and weight averaged molecular weights of which were estimated, by gel permeation chromatography (GPC), to be 5999.6 and 21287.7, respectively. The proanthocyanidin content in commercial grape skin extract products was determined by colorimetric analysis with vanillin-sulfuric acid to be around 60% (catechin equivalent value).

Keywords : grape skin extract, HPLC, anthocyanin, proanthocyanidin, tannin

I Introduction

Grapes (*Vitis* spp.) (Vitaceae) are a familiar fruit consumed globally. They are eaten raw and used to produce wines and juices. In previous phytochemical studies, their skins were reported to be composed of polyphenols such as the anthocyanin glucosides, *viz.* delphinidin, cyanidin, petunidin, peonidin, and malvidin, as well as gallic acid, (+)-catechin, (-)-epicatechin, quercetin, rutin, resveratrol, viniferin, and condensed tannins¹⁻⁶⁾. Other components include the polysaccharides⁷⁾, 1-triacontanol, β -sitosterol, and oleanolic acid³⁾. In the List of Existing Food Additives in Japan⁸⁾, grape skin extract is defined as "an additive obtained from the fruit skin of American grapes or grapes, having the polyphenol as its main constituent." Concerning the origin, manufacturing method, and essential qualities of these additives, the official

list states: "This additive is prepared from the pressed lees of the skin of Koshu, Chardonnay, or Riesling grape and all *Vitis labrusca* or *V. vinifera* varieties, by extraction with ethanol at lukewarm or room temperature".

Most existing food additives, which are officially registered under the Food Sanitation Act, are natural extracts containing various ingredients. However, the characteristic and effective components of the existing food additives are not always properly defined due to poor characterization of the ingredients in the respective raw material. Although the polyphenols from whole grapes have been exhaustively investigated, there is little detailed information on the chemical constituents of grape skin extract product. To ensure food safety, analysis and characterization of individual polyphenolic constituents, and establishment of analytical standards for developing national specifications is required.