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Pressurized low polarity water extraction of saponins from cow cockle seed

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Abstract

Pressurized low polarity water (PLPW) extraction of cow cockle seed was carried out to determine the effect of extraction conditions (temperature (125–175 °C), time (15–180 min)), and sample pre-treatment on saponin yield and composition. Accelerated solvent extractions (ASE) and ultrasonic extractions (USE) using water, methanol and ethanol (50%, 80%, 100%) were also carried out to determine the effect of extraction solvent and method on saponin recovery. A higher saponin yield was obtained by ASE compared to USE using pure and aqueous ethanol and methanol. The highest saponin yield, which was obtained by ASE using 80% ethanol, was used to calculate saponin recoveries. The saponin yield of PLPW extracts increased with extraction temperature and time. While only 33.2 wt% of total saponins was extracted from ground seeds at 125 °C in 3 h, 60.2 wt% was recovered in the first 15 min at 175 °C. Total extraction (1 h) of whole seeds yielded more saponins than ground seeds at 125–160 °C. Saponin concentration of the extracts was affected by the extraction solvent and method, sample pre-treatment and to a lesser extent by the time and temperature of PLPW extraction. The highest saponin concentration of PLPW extract was obtained at 125 °C using whole seeds (12%). The saponin composition of water extracts differed from that of aqueous ethanol and methanol extracts.

Keywords: *Vaccaria segetalis*; *Saponaria vaccaria*; Cow cockle seed; Saponins; Cyclopeptides; Subcritical water extraction; Ultrasonic solvent extraction; Accelerated solvent extraction; Bioactive components; Nutraceuticals

1. Introduction

Saponins are glycosides widespread in the plant kingdom and present in a few marine organisms such as starfish and sea cucumber (Hostettmann & Marston, 1995). They are categorized based on their structure containing a steroid or terpenoid aglycone attached to one or more sugar chains. Their structural diversity results in a number of physicochemical and biological properties with various industrial applications (Güçlü-Üstündağ & Mazza, in press). Mounting evidence on the biological activity of saponins such as cholesterol-lowering and anticancer properties (Kerwin, 2004; Oakenfull & Sidhu, 1990) has prompted research into investigation of new sources and processing methods for their commercial production (Dobbins, 2002; Muir, Paton, Ballantyne, & Aubin, 2002).

Cow cockle seed (*Vaccaria segetalis* Garcke, *Saponaria Vaccaria L.*, *Vaccaria pyramidata*) is an annual herb widespread in grain fields of the North Western United States and in the prairie provinces of Canada, Asia and Europe (Bailey, 1976; Mazza, Biliaderis, Przybylski, & Oomah, 1992). Although considered a weed in North America, cow cockle seed, known as Wang-Bu-Liu-Xing, has a prominent role in the traditional Chinese medicine (Sang, Lao, Chen, Uzawa, & Fujimoto, 2003). Its main uses include the promotion of diuresis and milk secretion, activation of blood circulation and the relief of carbuncle (Sang et al., 2003). Cow cockle seeds contain over 55%

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starch, 14% protein, 2–3% oil and around 2% saponins (Mazza et al., 1992). The bioactive components of cow cockle seeds include alkaloids, cyclopeptides, phenolic acid, flavonoids and steroids, in addition to saponins (Sang et al., 2003). Processing and properties of cow cockle seed starch, which has a small granular size, have been investigated (Brelsford & Goering, 1971), however, research on the bioactive constituents of cow cockle seed has largely been limited to isolation and identification of individual components (Jia, Koike, Kudo, Li, & Nikaido, 1998; Sang et al., 2003). Saponins isolated from cow cockle seed are listed in Fig. 1 and Table 1. Processing of cow cockle seed for the extraction/concentration of saponins and other bioactive components however has not been investigated.

Pressurized low polarity water (PLPW, also known as subcritical, hot, and superheated water) extraction involves the use of pressure to maintain water in the liquid state at temperatures above its normal boiling point. The higher temperatures thus achieved improves the mass transfer properties (faster diffusion rates) and decreases the polarity of water modifying its solvent power. For example, the dielectric constant of liquid water decreases with increasing temperature from ~80 at 25 °C to ~27 at 250 °C, which falls between those of methanol ($\varepsilon = 33$) and ethanol ($\varepsilon = 24$) (Hawthorne, Yang, & Miller, 1994). Increasing the temperature can also disrupt the solute–matrix interactions, and reduce the viscosity and surface tension of water improving the contact between the solvent and the solute (Richter et al., 1996). The high pressures used can further enhance the extraction of analytes trapped in matrix pores (Richter et al., 1996). Pressurized liquid extraction (PLE, Dionex trade name accelerated solvent extraction (ASE)), which involves the use of pressurized organic solvents, offers similar advantages.

The use of pressurized solvents at elevated temperatures can thus improve the efficiency of traditional processes resulting in shorter extraction time and lower solvent consumption. The dependence of solvent power/selectivity on temperature can be exploited to modify extract composition and for fractionation purposes.

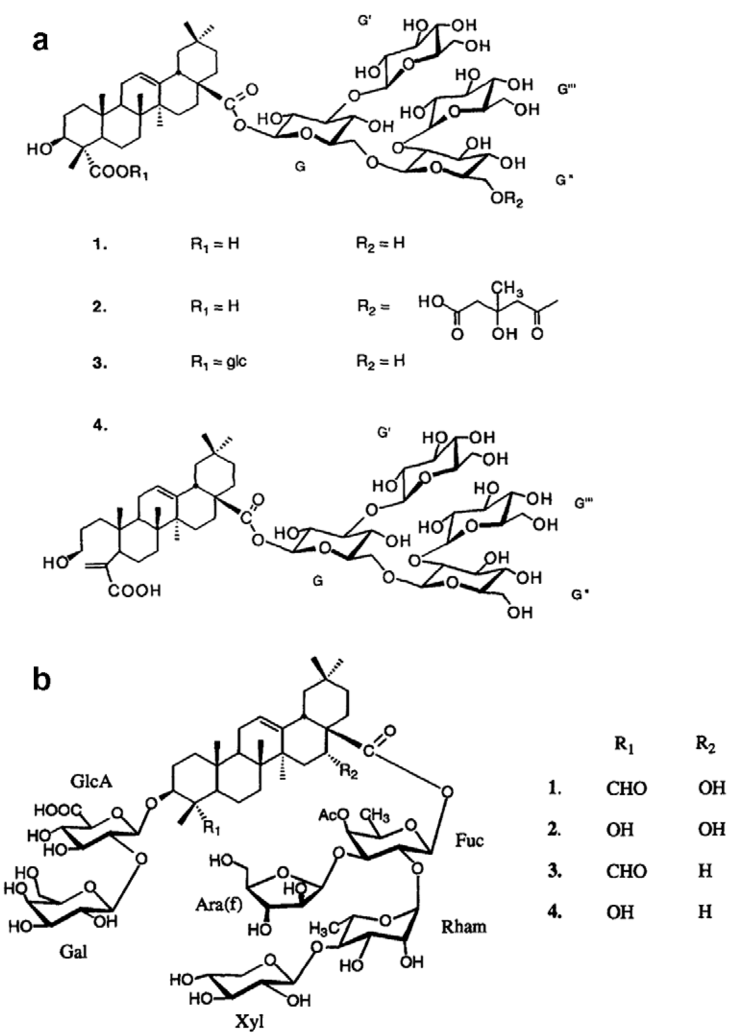

Fig. 1. Structure of cow cockle seed saponins (a) 1. Vaccaroside A, 2. Vaccaroside B, 3. Vaccaroside C, 4. Vaccaroside D; (b) 1. Vaccaroside E, 2. Vaccaroside F, 3. Vaccaroside G, 4. Vaccaroside H (Jia et al., 1998; Koike et al., 1998).