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Traditional rose liqueur – A pink delight rich in phenolics

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ABSTRACT

Chemical composition and colorimetric parameters of alcoholic liqueurs prepared from rose petals were evaluated by comparing the potential of three cultivars ('Amadeus', 'Colossal Meidiland' and 'Rosanna') and three traditional methods of preparation (fresh/air-dried petals extracted in 50% ethanol or aqueous sucrose syrup). Extraction was performed at room temperature for 2 weeks. High performance liquid chromatography/mass spectrometry was used to confirm the presence of 6 anthocyanins, 4 flavanols, 4 phenolic acids, 2 hydrolysable tannins and 31 flavonols in petal liqueurs. The highest concentrations of anthocyanins were determined in extracts from 'Amadeus' petals, followed by 'Colossal Meidiland' and finally, 'Rosanna'. The best extraction yields and optimal colour characteristics were achieved by ethanolic extraction of dry petals followed by fresh petal extraction in ethanol and, finally, extraction in sucrose syrup. Air-dehydration of 'Amadeus' petals prior to extraction in 50% ethanol yielded rose liqueur with the best all round characteristics.

1. Introduction

In recent decades interest in functional foods and beverages prepared from fresh and local ingredients has intensified (Harbourne, Marete, Jacquier, & O'Riordan, 2009). Some traditional products have been discovered anew and their potential health benefits are now at the centre of food research. Liqueurs and alcoholic drinks made from various fruit and herbal sources represent interesting marketable products that might also exhibit positive effects on human health, if consumed in moderation (Li & Beta, 2011; Mustafa et al., 2014). These products usually contain from 15 to 40% alcohol and are distinguished by their colour, taste, scent and other sensorial characteristics specific to the region and source of ingredients (Sokol-Letovska et al., 2014). Among the most popular alcoholic beverages are cherry, blackcurrant, raspberry, strawberry, chokeberry and sloe liqueurs, but other plant sources like roses have been utilized in the past to prepare a range of beverages (Stampar, Solar, Hudina, Veberic, & Colaric, 2006; Nour, Stampar, Veberic, & Jakopic, 2013; Senica, Stampar, Veberic, & Mikulic-Petkovsek, 2016).

Rosa sp. is one of the most widely utilized wild plants from antiquity to the present day. Traditionally, rose hips have been valued as a food source (processed into jams, preserves and other products) or for medicinal purposes (used fresh or dried) (Yildiz & Alpaslan, 2012; Demir, Yildiz, Alpaslan, & Hayaloglu, 2014). Rose leaves were also well-established in ancient Chinese medicine and in medieval European apothecaries as cures for the common cold and various inflammatory diseases (Coruh & Ercisli, 2010). Lastly, rose petals and young buds are valued as a fragrant natural additive for herbal infusions or, recently, vibrant delicacies in many high-end restaurants (Pires, Dias, Barros, & Ferreira, 2017). Hand-picked rose petals can also be used to prepare rose liqueurs with a delicate rosy colour and a unique aroma. Liqueur de Rose (France), Gulab (India) and, in Italy and Croatia, a similar drink is sold under the names Rosolio (Liquore Di Rose) and Rozulin.

A common method for preparing rose liqueur is soaking fresh petals in clear alcohol (Shen, Tseng, Chao, & Wu, 2007). Immersion in the alcohol or other liquids gradually 'extracts' the active compounds into the liquid. Phenolics are the predominant antioxidant compounds in these products, and their extraction depends on pre-treatment and soaking conditions. Several different procedures are utilized; most frequently, red or dark-pink fragrant rose petals are steeped in ethanol, left at room temperature for several weeks, strained through cheesecloth and supplemented with sugar syrup. Alternatively, petals are soaked in sugar syrup and alcohol is added at the end of the process.

Typically, rose liqueurs are made from fragrant old garden roses, but numerous hybrid cultivars have similar characteristics. These plants are cultivated mainly for their aesthetic appeal, but research has shown that modern hybrids accumulate comparable levels of beneficial compounds as hips from wild-growing roses (Cunja et al., 2016).

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Additionally, the intense colour of their flowers can be attributed to several classes of phenolics (Schmitzer, Veberic, Osterc, & Stampar, 2010), suggesting their suitability for rose liqueurs. We hypothesized that steeping petals from darker cultivars in different extracts would yield liqueurs with superior characteristics. The aim of this research was therefore, (1) to evaluate the potential of three modern rose hybrids for rose liqueurs, and (2) compare the efficiency of extraction using three different traditional extraction procedures as well as in pure ethanol. Except for colour quality analysis of rose liqueurs (Shen et al., 2007) and a recent study on rose hip alcoholic beverages (Sokol-Letovska et al., 2014), this is the first qualitative and quantitative analysis of secondary metabolites in rose petal liqueurs.

2. Materials and methods

2.1. Plant material

Fresh rose flowers were collected from the rose garden of Biotechnical faculty in Nova Gorica, Slovenia (45.95 °N, 13.64° E, elevation 103 m) in the morning hours of June 20th, 2016. A dark red climber 'Amadeus', red groundcover rose 'Colossal Meidiland', and pink climber 'Rosanna' (Supplementary material Fig. S1) were selected for analysis and only fully opened flowers (Schmitzer et al., 2010) were taken from three plants per cultivar, packed in paper bags and transferred to the laboratory. Only the petals were analysed and the entire (pooled) sample from an individual cultivar was divided between five treatments: (1) EEFP – fresh petals extracted in 97% ethanol, (2) EEDP – air dried petals extracted in 97% ethanol, (3) ESFP – fresh petals extracted in 50% aqueous ethanol, (4) SEFP – fresh petals extracted in sugar syrup and (5) ESDP – air dried petals extracted in 50% aqueous ethanol.

Petals for EEDP and ESDP were air dried in the laboratory at an ambient temperature of 25 (± 2) °C and 33% relative humidity for 72 h (Supplementary material Fig. S2). Masses of fresh petals were recorded immediately after transport to the laboratory and each day until day 3 to assess the decrease in water content. Portions of the samples were dried at 45 °C until a constant weight was achieved. Relative water content of rose petals was calculated as reported previously by Cunja, Mikulic-Petkovsek, Zupan, Stampar, & Schmitzer (2015).

2.2. Petal and liqueur colour assessment

Flower colour parameters were measured on fresh rose petals immediately after transport to the laboratory. The colour of air-dried samples was recorded daily for three days under uniform light conditions and orientation of the sample and the measuring device. Colour was measured in the middle of each petal (25 replicates per treatment) to ensure uniformity. A portable colorimeter was used for analysis of fresh/dry petal colour (CR-10 Chroma; Minolta, Japan). The colours are expressed as CIE $L^*a^*b^*$ (CIELab) values defined in a three-dimensional space. L^* represents the balance between the lightness (100) and darkness (0), a^* the balance between green (−60) and red (60); and b^* the balance between blue (−60) and yellow (60). The hue angle was calculated as arctan (b^*/a^*) and defines the basic colour: red (0° = 360°), yellow (90°), green (180°) and blue (270°) (Biolley & Jay, 1993).

The colour of rose liqueurs was assessed using Adobe Photoshop CS6 on five replicates per treatment. To acquire Lab colour space coordinates, individual samples were photographed in the laboratory under uniform conditions of light and camera settings. The photographs were analysed digitally, as an average value of 300 DPI (pixels/inch) in five replicates per photograph.

2.3. Rose liqueur preparation

Each treatment (EEFP, EEDP, ESFP, SEFP and ESDP) was prepared in five replicates. Prior to extraction, sugar syrup was prepared by heating 1000 mL of double distilled water containing 600 g of sucrose, and boiling the liquid for 5 min. The solution was cooled to room temperature and used in ESFP, SEFP and ESDP treatments.

EEFP and EEDP required extraction of 0.5 g of fresh and 0.2 g of air-dried petals in 3 mL of 97% ethanol, as described by Cunja et al. (2015). In ESFP, fresh petals (5 g) were extracted in 50% aqueous ethanol (60 mL) for 14 days. The extraction was performed in the dark and the samples were mixed daily. The extract was filtered through cheesecloth and the clear liquid supplemented with sugar syrup (1:1 v/v). ESDP was prepared using the same procedure, with the exception that air-dried rose petals (approx. 1 g) were used. SEFP rose liqueur was prepared by extracting fresh rose petals (5 g) in sugar syrup (60 mL) for 14 days. The extraction was also performed in the dark with daily mixing. At the end of the extraction, the liquid was filtered through cheesecloth and supplemented with 50% aqueous ethanol in 1:1 (v/v) ratio. The final volume, alcohol concentration and sugar contents of the rose liqueurs were identical for all treatments.

2.4. Determination of phenolic compounds using high-performance liquid chromatography coupled with mass spectrometry (HPLC/MS)

Extraction and determination of phenolic compounds was performed using HPLC/MS, as described by Cunja et al. (2015) with five replicates per treatment. Compounds were identified by comparing retention times, and spectral and fragmentation characteristics, and by adding authentic standards to the samples (spiking). The contents were calculated from peak areas and calibration curves for corresponding external standards. For compounds lacking standards, quantification was carried out using similar compounds as standards. Proanthocyanidin (PA) derivatives were quantified with the calibration curve of procyanidin B2, ellagic acid glycosides with ellagic acid, sinapic acid hexoside with sinapic acid, and compounds with a hexahydroxydiphenic (HHDP) moiety (hydrolysable tannins) with gallic acid. Eriodictyol was quantified with the calibration curve of naringenin and all kaempferol derivatives were expressed in equivalents of kaempferol-3-glucoside. The content of unidentified quercetin (Q) glycosides was assessed using the calibration curve of Q-3-galactoside. Cyanidin-di-glucoside, cyanidin-di-hexoside and cyanidin-hexoside-glucoside were quantified with cyanidin-glucoside and pelargonidin-di-glucoside with pelargonidin-glucoside. The content of individual compounds/phenolic classes are expressed in mg per 100 mL of rose liqueur or 97% ethanol.

2.5. Chemicals

Ethanol used in extraction of compounds was obtained from Sigma-Aldrich Corp. (St Luis, Mo, U.S.A). Purified water was obtained with Milli-Q Direct 8 system by Millipore (Merck KGaA, Darmstadt, Germany). For identification and quantification of phenolic compounds, the following standards were used from Sigma-Aldrich: ellagic acid, gallic acid, naringenin, Q-rhamnoside, Q-rutinoside, Q-xyloside, pelargonidin-3-glucoside and cyanidin-3-glucoside. Standards of procyanidin B2, catechin, (−)-epicatechin, sinapic acid, kaempferol-3-glucoside, Q-3-galactoside, Q-arabinofuranoside, and Q-glucoside were from Fluka (Fluka Chemie AG, Buchs, Switzerland).

2.6. Statistical analysis

Statistical analysis was performed with Statgraphics Plus 4.0 (Manugistics, Rockville, USA) using one-way analysis of variance (ANOVA). Duncan's multiple range test was used to determine differences between analysed parameters and phenolic groups/individual compounds among various procedures ($p < 0.05$). A two-factorial interaction between cultivar and procedure was calculated. Results are given in mean values, expressed in mg per 100 mL, with standard error.