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# Influence of hydrothermal process on bioactive compounds extraction from green coffee bean



### Adane Tilahun Getachew<sup>a,b</sup>, Byung Soo Chun<sup>a,\*</sup>

<sup>a</sup> Department of Food Science and Technology, Pukyong National University, 45 Yongso-ro, Nam-Gu, Busan 608-737, Republic of Korea <sup>b</sup> School of Chemical and Bioengineering, Addis Ababa Institute of Technology, Addis Ababa University, P.O. Box 385, Addis Ababa, Ethiopia

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#### ABSTRACT

This study reconnoiters the influence of subcritical water hydrolysis (SCWH) to recover bioactive substances rich soluble coffee extract (CE) from five types of raw coffee beans obtained from different geographical origins. The SCWH was conducted at a temperature of 180–220 °C and pressure of 30–60 bar. The extracts were evaluated for total phenolic content (TPC), total flavonoids content (TFC), individual phenolic acid content. Moreover, the extracts were evaluated for their antioxidant, antimicrobial and antihypertensive activity. The TPC and TFC ranged from 120.4 to 144.4 mg GAE/g and 15 to 43 mg/g catechin equivalent respectively. Chlorogenic acid found to be the dominant phenolic acid in all tested samples. The extracts showed high antioxidant and antimicrobial activity. Furthermore, the CE showed good *in vitro* antihypertensive activity with IC<sub>50</sub> values ranged from 1.989 to 2.562 mg/mL of lyophilized CE. CE from *Coffea canephora* showed better bio-functionality than that of *Coffea arabica* in all tested assays.

*Industrial relevance:* Recently, natural antioxidants and antimicrobial agents getting momentum in replacing their synthetic counterparts as the later have speculations to create health complications. Phenolic compounds from natural resource have a potential to replace synthetic preservative. However, extraction techniques to recover the phenolics from their sample matrix have significant impact on the yield, quality and economic feasibility in industrial application. In this study, we have showed a novel approach to extract bioactive compounds from raw coffee bean using subcritical water hydrolysis. The outcomes of the study demonstrated that, SCWH can be employed as green and an efficient extraction technique for production of soluble CE that might have a potential application in food and related industries.

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#### 1. Introduction

Coffee is one of the main traded commodity in the world economy just only behind to petroleum (Borrelli, Visconti, Mennella, Anese, & Fogliano, 2002). Two species account for virtually all coffee traded. *Coffea arabica* is the original coffee, native to the highlands of Ethiopia. *Coffea canephora* (generally known as robusta) originated in the low-land forests of West Africa (Linton, 2008). According to the World Coffee Organization (ICO, 2014), the world coffee consumption increased at an average annual growth rate of 1.9% over the last 50 years and expected to grow continually.

Epidemiological research suggests that moderate coffee drinking may help prevent several chronic diseases, such as diabetes and cardiovascular diseases (Higdon & Frei, 2006; Lecoultre et al., 2014). These beneficial effects have been attributed to the presence of phenolic acids more specifically chlorogenic acids (CGAs) (Shimoda, Seki, &

E-mail address: bschun@pknu.ac.kr (B.S. Chun).

Aitani, 2006) one of the most abundant class of compounds in coffee (Clifford, 2000). CGAs are formed within the coffee plant through the esterification of *trans*-cinnamic acids (caffeic, ferulic, and *p*-coumaric acids) with quinic acid, and may represent 6%–10% of the dry matter of the green coffee beans (Clifford, 1999; Ky, Noirot, & Hamon, 1997). To obtain the pleasurable aroma associated with coffee beverages green coffee beans need to be roasted (Del Castillo, Ames, & Gordon, 2002). The roasting process strongly modifies the final composition of coffee including CGAs (Mills, Oruna-Concha, Mottram, Gibson, & Spencer, 2013). It has been reported that roasting can contribute 42–99% loss of total CGA contents based on the roasting degree and coffee variety (Moon, Yoo, & Shibamoto, 2009). The very high loss of CGA during roasting process might compromise its potential benefits. So, an alternative method of obtaining valuable coffee bioactive compounds is needed.

Recently, subcritical water hydrolysis (SCWH) has become an increasing alternative technology in the extraction of bioactive compounds from natural resources (Herrero, Cifuentes, & Ibanez, 2006; Ramos, Kristenson, & Brinkman, 2002). Subcritical water refers to water at a temperature between 100 and 374 °C and at a pressure

<sup>\*</sup> Corresponding author at: Department of Food Science and Technology, Pukyong National University, 45 Yongso-ro, Namgu, Busan 608-737, Republic of Korea.

which is high enough to maintain the liquid state (Ramos et al., 2002). SCWH exhibits a number of advantages over conventional extraction methods such as solid–liquid extraction, microwave extraction, and ultrasound extraction. The important advantages of this method include its simplicity, reduced extraction time, higher quality of the extract, lower cost of the extracting agent, and being an environmentally friend-ly technique (Herrero et al., 2006).

Although SWE of bioactive compounds from coffee by-products has been reported, to best of our knowledge there is no documented report for the production of soluble coffee extracts (CE) from green coffee beans using this technique. Therefore, the aim of the present study was to produce soluble CE from green coffee bean obtained from different geographic origins using SCWH and to evaluate chemical and biological activities of the extracts.

#### 2. Materials and methods

#### 2.1. Materials

Five different coffee three *Coffee arabica* (from Ethiopia (Eth); Yigachefe, Guatemala (Gua), and Mexico (Mex)) and two *Coffee robusta* (from Brazil (Bra) and Vietnam (Vie)) were generously supplied by Global Soft Commodities GSC International Coffee®, Seoul, Republic of Korea.

O-phthalaldehyde (OPA), hippuryl histidyl leucine (*HHL*), caffeine, catechin, chlorogenic acid, caffeic acid, ferulic acid, gallic acid, hydrogen peroxide,  $\beta$ -carotene, linoleic acid, Folin-Ciocalteu reagent, Mueller-Hinton agar, Mueller-Hinton broth, ABTS (2,2-azinobis-3 ethyl benzothiazoline-6-sulphonic acid) and DPPH (2,2-diphenyl-1-picrylhydrazyl and acetonitrile were purchased from Sigma-Aldrich Chemical Co. (St. Louis, MI, USA). Iron (III) chloride 6-hydrate, trichloroacetic acid (TCA), L(+)-ascorbic acid, potassium ferricyanide, sodium carbonate, aluminum chloride, sodium hydroxide were purchased from Merck (Darmstadt, Hessen Germany). All other reagents used in this study were of high-performance liquid chromatography (HPLC) or analytical grade.

#### 2.2. Sample preparation

The coffee beans were manually sorted to remove defected beans and grounded and sieved to pass 710  $\mu$ m sieve. The mesh size of the powder was 25. The powder which passed through the sieve was transferred to airtight zip-lock and stored at 4 °C in a refrigerator until needed for further analysis.

#### 2.3. Subcritical water hydrolysis (SCWH)

Subcritical water hydrolysis was performed in a 200-cm<sup>3</sup> batch reactor made of 276 Hastelloy with temperature control (Fig. 1). Exactly 6 g of coffee powder was loaded into the reactor followed by 150 mL of distilled water. Then the reactor was closed and heated using an electric heater to the required temperature (180–220 °C) and pressures of (30–60 bar). The temperature and pressure in the reactor were controlled using a temperature controller and pressure gauge, respectively. The pressure was maintained to the required level using a nitrogen gas. The sample was stirred using a four-blade stirrer at 150 rpm. The times to reach the desired temperatures, 180 °C, and 220 °C were 20 and 33 min respectively. The hydrolysate samples from the reactor were collected after 10 min of extraction time, which was recorded after the desired temperature and pressure dusing Whatman nylon membrane filter (0.45  $\mu$ m) and stored at 4 °C until needed for analysis.

#### 2.4. Total phenolic content (TPC)

Total phenolic compounds were measured using the Folin Ciocalteau reagent as reported by Bravo, Monente, Juániz, De Peña and Cid (2013). Gallic acid was used as a standard, and the results were expressed as milligrams of GA per gram of raw coffee bean.

#### 2.5. Total flavonoid content (TFC)

The TFC of extracts were determined as described by Ozsoy, Can, Yanardag and Akev (2008)). Catechin was used as a standard. The



Fig. 1. Schematic diagram for subcritical water hydrolysis process.

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