PHENOLIC ANTIOXIDANTS EXTRACTION FROM HAZELNUT SHELLS

by

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Hazelnut shells are rich in potentially health-promoting phenolic antioxidants. Various novel extraction techniques, which are considered as efficient alternatives to conventional extraction methods, have been developed for the extraction of nutraceuticals from different feedstocks. However, limited research has been conducted on novel techniques for the highly effective extraction of phenolic antioxidants from hazelnut shells. From a commercial point of view, conducting research to extract phenolic compounds from hazelnut shells efficiently in terms of high extraction rate and yield, using newly developed techniques, is important and timely. Therefore, the goal of this project was to develop and optimize novel extraction methods for the production of phenolic antioxidants from hazelnut shells.

In this dissertation, effects of different extraction techniques, including conventional shaking bath extraction (SBE), and novel ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), and high pressure/temperature extraction (HPTE), and their extraction conditions on total phenolic content and antioxidant capacities in the extracts were investigated and compared. Phenolic composition of the extracts under different extraction processes were analyzed. The total phenolic content and antioxidant capacities of the phenolic extracts under the optimal conditions from Nebraska hybrid hazelnut shells also were evaluated and compared.

Results showed that hazelnut shells were rich in phenolic compounds with high antioxidant capacity. MAE could recover almost double the amount of total phenolics compared with SBE and UAE under their individual optimal extraction conditions, meanwhile, spending much shorter extraction time. Of these extraction methods, HPTE could recover the highest amount of phenolic compounds from hazelnut shells. Under the optimal HPTE conditions, the total phenolic content in the extracts was about 1.5 times higher than that of MAE, while the extraction time of HPTE was much longer than that of MAE and UAE using probe-type sonication, and the extraction temperature of HPTE was much higher than any other method. Among the tested Nebraska hybrid hazelnuts, cultivar 6-2 had the highest total phenolic content and antioxidant capacity.

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CHAPTER I

INTRODUCTION

1.1. Background

Hazelnuts, originated in the Mediterranean region, are an important commercial crop in many countries. The United States is the third largest hazelnut producer in the world, producing about 7% of the world's total hazelnuts, behind Turkey (~80%) and the European Union (~13%) (Huntrods, 2013). In the United States, commercial hazelnut production is mostly in the Willamette Valley in Oregon (Xu and Hanna, 2010b), and an average 37,625 tons were produced each year from 2008 to 2015 according to USDA (2015).

Hazelnuts are considered very healthy, and hold an important place among the types of dried nuts in terms of many of the important health categories because they are a rich source of unsaturated fatty acids, essential amino acids (mostly as arginine and leucine), dietary fiber, B vitamins, vitamin E, and minerals (Köksal et al., 2006). In the food industry, hazelnuts are currently eaten raw, roasted, blanched, minced, sliced, powdered, and pureed. Due to the distinctive flavor and nutritional value of hazelnuts, they are used as a premium ingredient in chocolates, caked foods, confectionary products, nut butter products, ice cream, and in meals and salads (Huntrods, 2013).

Based on different ways of consumption, the hazelnut market is segmented into two major categories: the in-shell and the shelled hazelnuts. In-shell hazelnuts are produced and marketed with the shell intact. However, the most common form of nuts used in the food-processing sector is the shelled nut (Ozdemir and Akinci, 2004).

Hazelnut shells represent more than 50% of the total nut weight (Xu and Hanna, 2009), and they are the major byproduct of hazelnut industry production. Hazelnut shells are composed of about

30% hemicellulose, 27% cellulose, and 43% lignin (Demirbas, 2006), so they are mainly utilized as a low-value heat source (Arslan et al., 2012). Other direct applications of the hazelnut shells in the production of activated carbon (Demirbaş et al., 2002; Kobya, 2004; Şayan, 2006) and bioplastic (Balart et al., 2016), and in pyrolysis (Di Blasi et al., 2015) have been carried out. Moreover, the conversion of hazelnut shells into useful chemicals such as methanol (Güllü and Demirbaş, 2001), hemicellulosic sugar (Arslan et al., 2012), reducing sugar (Uzuner and Cekmecelioglu, 2014), and furfural (Demirbas, 2006) have been reported. Recently, some effort has been made to utilize hazelnut shells as a low cost raw material for phenolic compounds extraction (Ciemniewska-Żytkiewicz et al., 2015; Contini et al., 2008; Shahidi et al., 2007; Xu et al., 2012).

Currently, commercial hazelnut cultivars in the USA are derived from the European hazelnuts and cultivated primarily in Oregon because European cultivars produce larger, higher quality nuts with thinner shells. However, the European cultivars cannot tolerate the harsh winters of the Upper Midwest, nor are they resistant to Eastern Filbert Blight (EFB). On the other hand, the native American species are cold-tolerant and EFB resistant, but the nuts are smaller and of less commercial value (Xu et al., 2012). Hybrid hazelnut shrub cultivars, which combine the superior qualities of the European hazelnuts and EFB resistance and cold hardiness of the native American hazelnuts, are emerging as a promising oil seed crop in the Upper Great Plains region of the USA. They require relatively low inputs and can be planted on marginal lands (Hammond, 2006). The results of a series of evaluations already completed by the Industrial Agricultural Products Center (IAPC) at the University of Nebraska - Lincoln (UNL) indicate that hybrid hazelnut kernels are potential feedstocks for food and value-added industrial applications (Xu and Hanna, 2010a; Xu and Hanna, 2010b; Xu and Hanna, 2011). Meanwhile, hazelnut shells, as the major byproduct of

processing, are rich in phenolic compounds that may serve as a potential source of natural antioxidants (Xu et al., 2012).

Plant-derived products contain a wide range of phytochemicals and phenolic compounds that possess substantial antioxidant and antiradical activities, anticarcinogenic (Kaliora et al., 2014), anti-inflammatory (Raso et al., 2001) and antimutagenic effects (Surh, 2003), and antiproliferative potential (de la Rosa et al., 2014). These phenolics provide protection against harmful effects of free radicals and are known to reduce the risk of certain types of cancer, coronary heart disease, cardiovascular disease, stroke, atherosclerosis, osteoporosis, inflammation, and other neurodegenerative diseases associated with oxidative stress (Shahidi et al., 2007). Thus, they are now used widely in the fields of biology, medicine, food, and cosmetics. Currently, many synthetic antioxidants, such as butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), propyl gallate (PG), and *t*-butylhydroxyquinone (TBHQ) are being used to retard the oxidation process, particularly in food systems. However, the application of synthetic antioxidants in food products is of high concern, and they are strictly regulated due to potential health hazards (Park et al., 2001). Consequently, the utilization of natural phenolic antioxidants, as alternatives, and the development of phenolic extraction methods have attracted global interest.

Extraction is a very important stage in the isolation, identification, and use of phenolic compounds (Lapornik et al., 2005). Phenolic compounds traditionally have been extracted by long maceration (Contini et al., 2008; Lu et al., 2011), heat reflux (Zhang et al., 2008), or Soxhlet extraction (Locatelli et al., 2010; Tyman et al., 1989). These extraction techniques are laborious and time-consuming. Moreover, these procedures require large amounts of toxic organic solvents, and thermo-labile compounds may suffer thermal decomposition (Luque de Castro and Garcıa-Ayuso, 1998). Due to the prescribed limitations of conventional techniques and the demands for more

efficient and economical processes of extraction in the food and chemical industries, alternative extraction techniques are being given more and more attention by researchers. Recently, various novel extraction techniques have been developed for the extraction of nutraceuticals from plants, including ultrasound-assisted extraction (UAE) (Roselló-Soto et al., 2015; Wang et al., 2013; Zhang et al., 2014), microwave-assisted extraction (MAE) (Simsek et al., 2012; Sun et al., 2016; Upadhyay et al., 2012), supercritical fluid extraction (SFE) (Roseiro et al., 2013; Solana et al., 2016), subcritical water extraction (SWE) (Etoh et al., 2010; Rangsriwong et al., 2009; Singh and Saldaña, 2011), high hydrostatic pressure (HHP) extraction (Corrales et al., 2008), high pressure/temperature extraction (HPTE) (Casazza et al., 2012; Yang et al., 2013), pyrolytic distillation (Ma et al., 2011; Wei et al., 2010a; Wei et al., 2010b), pulsed electric field (PEF) extraction (Boussetta et al., 2014; Boussetta et al., 2012), and high voltage electrical discharge (HVED) extraction (Boussetta et al., 2009a; Boussetta et al., 2009b; Boussetta et al., 2012). Compared with traditional extraction methods, the newly developed techniques are more efficient in terms of higher yields, shorter extraction times, lower amounts of extraction solvents, and sometimes lower temperatures.

Although hazelnut shells are rich in phenolic compounds, limited research has been conducted to extract phenolic compounds from hazelnut shells using those novel extraction techniques for the high efficiency production of phenolic compounds. The goal of this project was to develop and optimize novel extraction methods (UAE, MAE, and HPTE) for the production of phenolic compounds from hazelnut shells. The long-term goal is the application of the optimized process to increase the value of hazelnuts.

1.2. Objectives

Overall, this dissertation research was carried out to explore the extraction techniques for phenolic antioxidants production from hazelnut shells, with the specific objectives to:

1) evaluate the phenolic content and composition, and antioxidant activities of extracts from hazelnut shells by conventional shaking bath extraction;

2) develop an ultrasound-assisted extraction method with an ultrasound bath for the phenolics extraction from hazelnut shells;

3) develop an ultrasound-assisted extraction method with a probe sonicator for the phenolics extraction from hazelnut shells;

4) develop a microwave-assisted extraction method for the phenolics extraction from hazelnut shells; and

5) develop a high pressure/temperature extraction method for the phenolics extraction from hazelnut shells.

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