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Effect of Drying and Roasting to Antioxidant Property and Stability of Dried Roasted Walnut *(Juglans Regia)*

N. P. Minh[1,*], P. T. L. Pha[2], N. T. T. Trang[3], B. N. Y[4]

[1]*Faculty of Food Technology - Biotech, Dong A University, Da Nang City, Vietnam*
[2]*Can Tho University, Can Tho City, Vietnam*
[3]*Le Van Tam Secondary - High School, Nga Nam District, Soc Trang Province, Vietnam*
[4]*An Giang University, An Giang Province, Vietnam*

Abstract.

Walnut is one of the important nut fruits in the world with essential component of high nutrition values. Walnut plant (*Juglans regia*) is a medicinal plant with different properties having great therapeutic potential in the traditional medicine. *Juglans regia* L. is a natural product of high economic interest to the food industry and is very popular and largely consumed as royal food globally and valued for its nutritional, health, and sensory attributes. Walnut kernels are nutrient-rich and an excellent source of antioxidant products mainly owing to their high levels of oil, high total phenolic concentration, and total antioxidant capacity. Developing effective drying and roasting for walnuts (*Juglans regia* L.) is a major postharvest processing concern in the nut industry. An attempt was to identify the effect of temperature in drying, roasting and preservation to antioxidant (total phenolic and total flavonoid) in the dried roasted walnut (*Juglans regia*). Results demonstrated that drying temperature (45 ^{o}C) and roasting (135 ^{o}C in 3 min) was adequate to preserve it for 12 months in PET/AL/PE bag without deterioration.

Keywords: *Walnut, drying, roasting, total phenolic, total flavonoid*

I. INTRODUCTION

Walnut (*Juglans regia* L.) a species belonging to the Juglandaceae family, is a medicinal plant known more particularly for its active ingredient, Juglone, the latter confer to the plant several therapeutic virtues (J. Liu, 2008). *J. regia* is the sole species within the subgenus Juglans, and is characterised by a four-celled nut, a husk which separates from the nut at maturity, and seedlings which exhibit two rows of scale buds immediately above the cotyledons and below the spirally-arranged compound leaves. J. regia is medium to large tree (up to 30 m tall in natural conditions) with a spreading crown. The leaves are compound and composed of 7 to 9 leaflets, which have prominent, herring-bone venation. Leaflets are ovate, with pointed tips and smooth margins. Nuts develop in groups of one to three on shoot tips. A green, fleshy husk surrounds the nut, which splits irregularly at maturity. The husk is easily separated from the nutshell. The shell is rough, wrinkled or furrowed, and thin. Nuts are ovoid to round, 1.25 to 5.00 cm in diameter, and contain two kernels separated by a thin, papery central plate extending from the inner layer of the shell. Nut production starts usually at age 4 to 6 years. Walnut (*Juglans regia*) contains tannins, flavonoids, alkaloids, free quinones, anthocyanins and saponins, which could be responsible for the biological properties (Bennacer Amel, Cherif Hamida Saida, 2016). While this plant having high antioxidant capabilities, walnuts are composed of many chemical compounds such as ascorbic acid, flavonoids, quercetin, and caffeic acid. Walnuts contain important antioxidants that are mainly tocopherols and phenolic compounds (Kornsteiner et al., 2006; Pereira et al., 2008). They inhibit *in vitro* plasma and LDL oxidation (Anderson et al., 2001; Chen & Blumberg, 2008).The antioxidant activity in 1-serving portion of fresh or dry walnuts is equivalent to that in almost 2-serving portions of black tea (Iskender Arcan, Ahmet Yemeniciog˘lu, 2009). Experimental studies have shown that walnuts reduced blood glucose and lipids and also decreased blood pressure. They have antioxidant, antidiabetic, antimicrobial, and liver-protective properties. The use of walnuts in traditional medicine and review of experimental studies demonstrated the presence of multiple, effective, and useful compounds which may provide the opportunity for the production of lipid-lowering, antidiabetes, and liver protective drugs (Hamdollah Delaviz et al., 2017). Walnut is a valuable medicinal plant with a potency to cure various diseases in traditional medicine including helminthiasis, diarrhea, sinusitis, stomach ache, arthritis, asthma, eczema, scrofula, skin disorders, diabetes mellitus, anorexia, thyroid dysfunction, cancer and infectious diseases (NishaPanth et al., 2016). Natural polyphenols are important compounds present in walnut with valuable properties that have been studied for the treatment of inflammation, cancer or anti-ageing effect.

Juglans regia is considered to treat a variety of health complaints traditionally, including cancer, inflammation, diabetes, antiradicalar, hyperhidrosis, antidiarriec, prostate, antiradicalar, and cardiovascular disorders (Girzu, M. et al., 1998; Mouhajir, F. et al., 2001; Vaidyaratnam, P. S. V., 2005; Baharvand-Ahmadi, B. et al., 2016). The extracts from *J. regia* nut inhibited oxidative damages (Isabel, F. A. et al., 2008; Carvalho, M. et al., 2010; Sharma, P. et al., 2013; Zhao, M. H. et al., 2014), inflammation (Papoutsi, Z. et al., 2008; Hosseinzadeh, H. et al., 2011), tumor growth (Negi, A. S. et al., 2011),antiwrinkle, and photoageing (oshan, D. S.; Singh, S. K., 2013).Kernels as a dietary food, against diabetes, hypoxia, some skin diseases, and inflammation (Tsao, R., 2010; Ram, S., 2013); leaves as antidiarrheals, anthelmintic, depurative and also mixed with stored-grains as an insecticide and fungicide (Negi, K. S.; Kanwal, K. S., 2009). Stem bark as an astringent, anthelmintic, depurative (Cosmulescu, S.; Trandafir, I., 2011), bactericide, diuretic, digestive, laxative, stimulant, detergent, and insecticidal (Espin, J. C. et al., 2000).*Juglans regia* L. shell is reported for polishing gun-casings, jewelry, and metal material and is used as media to

separate water and crude oil (Srinivasan, A.; Viraraghavan, T., 2008).

There were several researches mentioned to Walnut production. Selected drying conditions and storage period and quality of walnut selections were investigated (M. A. Koyuncu et al., 2003). Effect of boiling and traditional roasting on the nutritional, antinutritional, and antioxidant properties of African walnuts seeds (Arinola & Adesina, 2014); impact of processing on the nutrient content, vitamin, and mineral composition of African walnuts (Okonkwo & Ozoude, 2014); effect of cooking on phenolic content and antioxidant properties of African walnuts seeds (Ademiluyi et al., 2015). A research developed hot air-assisted RF (HARF) drying protocols for in-shell walnuts (Bo Zhang et al., 2016). Effect of intermittent oven drying on lipid oxidation, fatty acids composition and antioxidant activities of walnut was verified (MaorunFu et al., 2016). Comparative analyses of three dehydration methods on drying characteristics and oil quality of in-shell walnuts were conducted (Xu Zhou et al., 2018). The effect of boiling and roasting on the lipid quality, proximate composition, and mineral content of African walnut seeds (*Tetracarpidium conophorum*) was assessed (Fabrice Tonfack Djikeng et al., 2018).

Fresh walnut contains a moisture content of about 35-40 %(dry basis). They are susceptible to germination and decay due to their high water and oil contents at harvest, which would result in poor quality stability and short shelf life of walnut products (Ma, Y. P. et al., 2013). Drying and roasting of walnuts immediately after harvest is critical to preserve their quality. Fresh walnuts must be dried to a moisture content of 8.0 and 5.0% on a dry weight basis (d.b.) of the whole walnuts and kernels, respectively, for long time storage in an ambient environment (Amaral, J. S. et al., 2003).

. So the objective of the present study was to identify the effect of temperature in drying, roasting, preservation to antioxidant (total phenolic and total flavonoid) in the dried roasted walnut (*Juglans regia*).

Figure 1. Walnut (*Juglans regia*)

II. Materials And Method

2.1 Material

Walnuts (*J. regia* L.) with green husks were harvested in in Lam Dong province, Vietnam. All walnuts used for drying and roasting were obtained from the same batch. After dehulling, only the uniform size nuts were used in this study. To ensure the uniform size of samples used in the experiment, the walnuts were prescreened before being transported to the laboratory. Immediately after arriving, the walnuts were hulled and then washed with clean tap water. After cleaning, the nuts were directly dried and roasted in different conditions. Finally, the samples were packed and vacuum-sealed into PET/AL/PE bags. All packaged samples were stored in ambient temperature (28^{o}C) until utilized for experiments.

2.2 Researching procedure

2.2.1 Effect of drying temperature to total phenolic and total flavonoid content in dried walnut (Juglans regia)

In order to verify the effect of drying temperature to total phenolic and total flavonoid content in dried walnut (*Juglans regia*), the total phenolic (mg GAE/ 100g) and total flavonoid (mg QE/100 g) content will be analyzed before drying)fresh Walnut (*Juglans regia*)) and after drying in different drying temperature (35^{o}C, 40 ^{o}C, 45 ^{o}C and 50 ^{o}C).

2.2.2 Effect of roasting conditions to total phenolic and total flavonoid content in the dried roasted walnut (Juglans regia)

After completion of drying treatment, the dried nuts were subjected to roasting at different conditions (125 ^{o}C for 4 min, 135 ^{o}C for 3 min, and 145 ^{o}C for 2 minutes). The total phenolic (**mg GAE/ 100g**) and total flavonoid (mg QE/100 g) will be analyzed to verify the appropriate roasting condition.

2.2.3 Changes in lipid oxidation in dried roasted walnut (Juglans regia)

The dried roasted walnut (*Juglans regia*) was kept in PET/AL/PE bag in ambient temperature. Acid value (mg/g), peroxide value (meq/kg), saponification value (mg/g), Iodine value (g/kg) will be analyzed in 3 months interval for 12 months.

2.3 Phytochemical analysis

Total phenolic content (mg GAE/ 100g) was analyzed by using Folin–Ciocalteu method (Vuong, Q. et al., 2014). Total flavonoids content (mg quercitin equivalents/100 g) was determined on the basis of formation of flavonoid-aluminum complex by using spectrophotometric method (Lamaison, J. L. et al., 1991). Acid value (mg/g), peroxide value (meq/kg), saponification value (mg/g), Iodine value (g/kg) SV were calculated according to the AOAC (2000) standard method.

2.4 Statistical analysis

The Methods were run in triplicate with three different lots of samples. Data were subjected to analysis of variance (ANOVA) and mean comparison was carried out using Duncan's multiple range test (DMRT). Statistical analysis was performed by the Statgraphics Centurion XVI.

III. Results & Discussion

3.1 Effect of drying temperature to total phenolic and total flavonoid content in dried walnut *(Juglans regia)*

Drying time and temperatures have important effects on the product quality. Among the common plant foodstuffs and especially nuts, walnuts rank at the top of the scale for their antioxidant capacities, as determined by both the ferric reduced power assay and free radical scavenging activity tests, since they exhibit high total phenolic content (Wanyo, P. et al., 2016). Walnuts are rich in phenolic compounds and have higher TPC than other nuts (Kornsteiner et al., 2006; Reddy et al., 2010). Phenolic compounds, which

impact both the sensory properties and the bioavailability of plant foods, are significant bioactive compounds of walnuts. They have become very popular recently, in view of their relationship with nutrition and health. A fistful of crushed walnuts have much more phenolic compound than do a milky chocolate bar, a glass of red wine and apple juice (Anderson et al., 2001). The contribution of phenolic compounds and a fatty acid profile, identified by related studies, revealed that the consumption of walnuts reduces LDL cholesterol oxidation and plasma lipid peroxidation and, remarkably, causes increases in plasma antioxidant capacity (Anderson et al., 2001; Griel & Kris-Etherton, 2006).

In order to verify the effect of drying temperature to total phenolic and total flavonoid content in dried walnut (Juglans regia), the total phenolic (mg GAE/ 100g) and total flavonoid (mg QE/100 g) content will be analyzed before drying (fresh Walnut (*Juglans regia*)) and after drying in different drying temperature (35^{o}C, 40 ^{o}C, 45 ^{o}C and 50 ^{o}C). From table 2, the Walnut (*Juglans regia*) should be dried at below 45^{o}C to maintain the highest amount of total phenolic and total flavonoid content

Table 1. The total phenolic (mg GAE/ 100g) and total flavonoid (mg QE/100 g) content in dried walnut (*Juglans regia*) by the effect of drying temperature (^{o}C)

Parameter	Fresh walnut (*Juglans regia*) before drying	Dried walnut (*Juglans regia*) by the effect of at drying temperature (^{o}C)			
		35	40	45	50
The total phenolic (mg GAE/ 100g)	62.38±0.01^{a}	53.31±0.01^{b}	53.27±0.02^{bc}	53.25±0.01^{c}	52.073±0.02^{d}
Total flavonoid (mg QE/100 g)	71.12±0.03^{a}	65.48±0.02^{b}	65.11±0.00^{bc}	65.03±0.01^{c}	62.36±0.02^{d}

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant (α = 5%).

Table 2. Effect of roasting conditions on tocopherol content in the roasted dried Walnut (*Juglans regia*)

Roasting conditions	125 ^{o}C for 4 min	135 ^{o}C for 3 min	145^{o}C for 2 min
The total phenolic (mg GAE/ 100g)	50.34±0.02^{b}	51.43±0.00^{a}	49.05±0.03^{c}
Total flavonoid (mg QE/100 g)	62.17±0.03^{b}	63.78±0.02^{a}	61.12±0.01^{c}

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant (α = 5%).

Table 3. Acid value (mg/g), peroxide value (meq/kg), saponification value (mg/g), Iodine value (g/kg) of dried roasted walnut *(Juglans regia)* by preservation

Storage time (months)	Acid value (mg/g)	Peroxide value (meq/kg)	Saponification value (mg/g)	Iodine value (g/kg)
0	0.21±0.03^{c}	0.17±0.03^{c}	31.74±0.01^{c}	3.02±0.00^{c}
3	0.33±0.00^{bc}	0.24±0.01^{bc}	93.38±0.01^{bc}	4.11±0.03^{bc}
6	0.47±0.01^{b}	0.38±0.02^{b}	121.04±0.00^{b}	5.78±0.02^{b}
9	0.53±0.02^{ab}	0.46±0.00^{ab}	195.48±0.02^{ab}	6.05±0.03^{ab}
12	0.68±0.03^{a}	0.53±0.03^{a}	224.16±0.03^{a}	6.55±0.01^{a}

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant (α = 5%).

Beygi et al. (2009) determined drying characteristics of walnuts during convective drying at air temperatures of 32 °C and 43 °C and two air velocities of 1 and 3 m/s. A new pilot size walnut indirect solar batch type dryer was designed and fabricated. The effect of crucial factors affecting drying time namely: drying air temperature (T1: 37^{o}C; T2: 39^{o}C and T3: 41^{o}C), drying air flow rate (F1: 0.065 m3 /s, F2: 0.075 m3 /s, F3: 0.09 m3 /s) and bed depth (D1: 2 layers, D2: 4 layers and D3: 6 layers) were evaluated. The experimental results showed that, the effect of above factors on walnut moisture loss are significant. With increasing drying air temperature, drying air flow rate and number of layers, the amount of average moisture loss decreased from 37% (d.b) to 9% (d.b) at 3 hrs. Performance of dryer was determined at temperature 41^{o}C, air flow 0.09m3 /s, 2 layers depth (Ghatrehsamani S.H. and Zomorodian A., 2012). A research developed hot air-assisted RF (HARF) drying protocols for in-shell walnuts. Results showed that an electrode gap of 18.0 cm combined with hot air temperature of 50°C provided an acceptable heating rate and stable sample temperatures during RF drying. Total drying times to reduce the whole walnut moisture content from 20 to 8.0% on dry basis required 240 and 100 min using hot air (HA) drying and HARF drying, respectively (Bo Zhang et al., 2016). Air-assisted radio frequency drying and hot air drying had little effect on the antioxidant capacity and total phenolic concentration of walnuts during the drying process and storage (Xu Zhou et al., 2018).

3.2 Effect of roasting conditions to total phenolic and total flavonoid content in the dried roasted walnut *(Juglans regia)*

During thermal processing, eventhough antinutritional components are reduced or eliminated; heat has a detrimental effect on the nutritional and functional properties of foods (Kanu et al., 2015). Then, the thermal treatment of walnuts can lead to chemical changes that can affect its nutritional value and the quality of its lipids. During roasting of the seeds, high temperatures can facilitate lipid oxidation and nonenzymatic browning reactions, which can reduce the nutritional value of foods, causing the loss of essential fatty acids, essential aminoacids and carbohydrates. The amount of vitamins can also be reduced as well as the proteins digestibility

(Cuvelier & Maillard, 2012). Additionally, these chemical alteration reactions may generate toxic compounds in edible seeds and the derived products, which can be harmful for the consumers (Djikeng et al., 2017).

Due to the strong bitter taste, walnut seeds are usually consumed after roasting, which also contributes to the elimination of antinutrients. After completion of drying treatment, the dried nuts were subjected to roasting at different conditions (125 ^{o}C for 4 min, 135 ^{o}C for 3 min, and 145 ^{o}C for 2 minutes). The total phenolic (mg GAE/ 100g) and total flavonoid (mg QE/100 g) will be analyzed to verify the appropriate roasting condition. Results were elaborated in table 2. Walnut (*Juglans regia*) should be roasted at 135 ^{o}C for 3 min to preserve the total phenolic and total flavonoid at utmost level.

The effect of boiling and roasting on the lipid quality, proximate composition, and mineral content of African walnut seeds (*Tetracarpidium conophorum*) was assessed. Results indicated that the quality of walnut oil significantly ($p < .05$) reduces with the treatments. Oils extracted from *DBWN* 60 min (Dried and boiled walnuts 60 min) and *FBWN* 60 min (Boiled fresh walnuts 60 min) were the most altered. The proximate composition and mineral content of walnut seeds was also significantly affected ($p < .05$) by the treatments. This study reveals that, thermal processing has significant effects on the nutrients and quality of lipids of walnut oil. *DTRWN* 60 min (Dried and traditionally roasted walnuts 60 min), *DORWN* 60 min (Dried and oven roasted walnuts 60 min), and *TRFWN* 30 min (traditionally roasted fresh nuts 30 min) are the best methods for cooking walnut because they preserve the quality of its lipids and some of the nutrients (Fabrice Tonfack Djikeng et al., 2018).

3.3. Changes in lipid oxidation in dried roasted walnut *(Juglans regia)*

The acid value (mg/g) can be used to express the value of free fatty acids forming during the long storage periods due to oil hydrolytic rancidity. If the lipase activity was high, the acid values rose rapidly. The peroxide value (meq/kg) can be considered as a sign of oxidative rancidity, reflecting the value of hydroperoxide as fatty acid oxidation takes place. The saponification value (mg/g) is an important indicator of the liquidity and the hydrophilicity of the oil. The Iodine value (g/kg) is often used to determine the amount of unsaturation in fatty acids, as this unsaturation is in the form of double bonds (C=C), which react with iodine compounds. The higher iodine value, the more C=C bonds are present in the oil (Xu Zhou et al., 2018).

The dried roasted walnut (*Juglans regia*) was kept in PET/AL/PE bag in ambient temperature. Acid value (mg/g), peroxide value (meq/kg), saponification value (mg/g), Iodine value (g/kg) will be analyzed in 3 months interval for 12 months. From table 3, the roasted dried walnut (*Juglans regia*) could be stable for 12 months of storage. Christopoulos and Tsantili (2011) reported that the level of TPCs in walnut kernels decreased progressively with advanced storage time.

In another report, quality losses in the shelled walnuts were greater than quality losses in the unshelled walnuts. Walnuts removed from their hulls and dried under sun can be stored under ambient conditions (21 ± 1C and 50–65 RH) and retain acceptable quality for 12 months (M. A. Koyuncu et al., 2003). Meanwhile, Labuckas et al. (2011) and Vanhanen et al. (2006) showed that walnut oils remained stable or slightly fluctuated in a narrow range, which mainly due to the protective effect of the antioxidant compounds.

IV. CONCLUSION

Juglans regia L. is a good source of flavonoids, polyphenols, flavonols, carbohydrates, fatty acids, cardiac glycosides, steroids, minerals, tannins, protein, dietary fiber, melatonin, plant sterols, α-tocopherol, folate, tannins, vitamin A and C, and vitamin E family compound. *J. regia* possess significant antioxidant and antimicrobial activity. Drying was an energy intensive operation of some industrial application. The most important of drying was the reduction of moisture content to a required level to prevent spoilage, allowing safe storage and prevent microbial development or other harmful reaction which might lead reduction of chemical quality. It should be reduced to 4-8 % by drying in order to maintained the oil content. Phytochemical characteristics of dried roasted walnut (*Juglans regia*) were significantly affected by drying and roasting condition. Dried roasted walnut can be used as functional food.

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