

The Combination of Heat Pump Dryer and Vacuum Microwave Dryer on Production of High Bioactive Compound Mulberry Leaf Powder

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Abstract

The objective of this research was to produce mulberry leaf powder that had high bioactive compounds and low production cost. From the physicochemical study of difference mulberry leaf maturity levels, mature leaf had the highest amount of 1-Deoxynojirimycin (1-DNJ) and antioxidant activity (FRAP method). All maturities had slightly different on bioactive compounds and antioxidant activities. In practical, could be used mixed all mulberry leaf maturities as raw material for mulberry leaf powder production. From traditional hot air oven drying studying, it was found that drying at 50-60 °C 5 hours drying time could provide high bioactive compound mulberry leaf powder. From combination of heat pump drying and vacuum microwave drying, primary dewatering preparation of mulberry leaf to 50% moisture content of fresh leaves could be done by drying in heat pump dryer at 60 °C for only 15 minutes. For final dewatering step, it could be done by drying in vacuum microwave dryer at 4,800 W magnetron power for only 20 minutes. This combination method provided 27.24% of production yield from fresh mulberry leaves. Its electricity cost was 11.55 baht/kg of mulberry leaf powder which was lower than traditional hot air oven drying about 10 times. If the cost of mulberry leaf at front of factory was 10 baht/kg, the production cost (mulberry leaf, electricity and equipment depreciation cost) was 193.34 baht/kg of leaf powder. This product had 5.29 μmol GAE/g (dried basis) of phenolic compounds and 786.00 mg/100g sample of total chlorophylls. For antioxidant activities which were assayed using DPPH, ABTS and FRAP methods, there were 8.10 μmol of trolox/g dried basis, 16.23 μmol of trolox/g (dried basis) and 54.59 μmol of Fe²⁺/g (dried basis), respectively. From this study, selected mulberry leaf powder processing had high potential for commercial production as supplementary food which could be directly or encapsulated consumed.

Keywords: Mulberry leaf powder, Heat pump dryer, Vacuum microwave dryer, Primary dewatering, Final dewatering

1. Introduction

Mulberry (*Morus alba* L., family of Moraceae) leaves, one of the most widely used in traditional Chinese medicines, have attracted much attention because of their multiple pharmacological effects, such as anti-diabetic, anti-inflammation, anti-cancer, and so on (Andallu et al., 2001, Andallu and Varadacharyulu, 2007). Previous studies have confirmed that various alkaloids, including 1-deoxynojirimycin (DNJ), 1,4-dideoxy-1,4-imino-D-arabinitol (DAB), and 1,4-dideoxy-1,4-imino-D-ribitol (DRB), could have beneficial physiological effects on diabetes (Kimura et al., 2004). In recent years, the antidiabetic activity of crude polysaccharide extract (Chen et al., 2011 and Wang et al., 2010) and the mixture of DNJ and polysaccharides (Li et al., 2013) from mulberry leaves have been reported. Mulberry leaves contain many bioactive compounds like as moranolin, moran, flavones, 2-arylbenzofuran, carotenoids, γ -aminobutyric acid, polyphenols which play a significant role in hypoglycemic activity, anticancer action, antioxidants action, anti-inflammatory actions. There are many polyhydroxylated alkaloids which were extracted like as piperidine, pyrrolidine, indolizidine. Due to structural similarity to sugars, most of the polyhydroxylated play as antihyperglycemic which is inhibitory activity against glycosidase (Bajpai et al., 2014).

Drying is an important process for preserving agricultural products. Drying reduces the moisture content to a level which allows safe storage over an extended period. Heat pump dryer (HPD) which consisting of a conventional drying chamber with an air circulation system and the usual components of an air-conditioning refrigeration system are extensively used. Air is dehumidified by the evaporator (cooling section of the refrigeration cycle) and reheated by the heat pump condenser. Commonly, heat pump dryers can operated in the range 35-60°C. A heat pump dryer is used as alternative to dry foods or biologically active products because it consumes less energy and does not cause pollution. This method also involves low relative humidity, low temperature, and good quality of product (Ahmad et al., 2016)

Vacuum microwave dryer (VMD) is a novel alternative drying equipment an uniform electromagnetic field for rapid drying. Microwave provides a rapid heating process and can selectively target only water in material. Reduced pressure lowers the boiling point of water and can accelerate vaporization of moisture in these materials during the drying process. VMD has been used to dry many types of materials. VMD, both in terms of energy efficiency and quality of the dried product, has many advantages. The combination of microwaves and vacuum can greatly improve energy efficiency and product qualities. Thereby, microwave drying combined with vacuum can increase drying rate and reduce drying time (Huang-Feiet al., 2018).

In this study, combination of heat pump drying and vacuum microwave drying for mulberry leaf drying was compared to a traditional hot air drying. Heat pump drying used for primary dewatering stage and vacuum microwave drying was used for final dewatering stage. Bioactive compounds and production cost of mulberry leaf powder were also considered and discussed.

2. Materials and Methods

2.1 Study on effect of leaf maturities on physicochemical compositions and bioactive compounds of fresh mulberry leaves

Mulberry leaves (Burirum 60 variety) were harvested from The Queen Sirikit Department of Sericulture (Chiang Mai, Thailand). Leaves were classified into three groups based on maturity levels; young (the first of fourth leaves from the tip), mature (the fifth to eighth leaves), and old (leaves below the ninth leaf).

The physicochemical compositions of fresh mulberry leaves were determined as follows:

- Size measurement by Vernier caliper using AOAC (2000) method
- Weighting by digital scale
- Color value determination by Minolta Chroma Meter
- Proximate analysis for moisture, fat, protein, crude fiber, ash and carbohydrate using AOAC (2012) method.

The bioactive compounds of fresh mulberry leaves were determined as follows:

- Total phenolic compounds by Folin-Ciocalteu method (modified from Cai et al., 2013)
- Total chlorophylls using AOAC (2000) method
- 1-Deoxynojirimycin (DNJ) using LC-MS/MS
- Antioxidant activities were determined 3 methods: DPPH radical scavenging activity (modified from Brand et al., 1995), ABTS radical scavenging capacity (modified from Cai et al., 2013) and ferric reducing antioxidant potential (FRAP) (modified from Benzie and Strain et al., 2013)

The experimental design was Completely Randomized Design (CRD) with 3 replications. The data obtained from the experiments were processed with the Analysis of Variance (ANOVA) and mean compared using Duncan's new multiple range test ($P \leq 0.05$). The maturity stage that had high bioactive compounds and high amount of quantities for commercial production scale was selected for further study.

2.2 Study on the effect of heat pump dryer (HPD) compare with vacuum microwave dryer (VMD) on bioactive compounds

The raw mulberry leaves were soaked and washed with chlorinated water (200ppm) for 10 minutes and span-out to remove excess water for 5 minutes. Pretreated mulberry leaves were heat pump drying at temperature 60°C compare with vacuum microwave drying at 4,800 W level of magnetron. During drying, the vacuum was fixed at 0.8 mmHg. Drying time was recorded after drying until less than 6% moisture content. After drying, dried leaves were crushed and 50 mesh sieved. Mulberry leaf powder was determined moisture content, yield of production, total phenolic compounds and antioxidant activities (using DPPH, ABTS and FRAP methods). The electricity consumptions were compared.

The experiment was CRD with 3 replications. The data were statistical analyzed using ANOVA and the means were compared using Duncan's new multiple range test ($P \leq 0.05$). The suitable pre-drying method was selected from the

2.3 Study on the combination method for mulberry leaf drying

The pre-dried mulberry leaves from 2.2 were final dried using the vacuum microwave dryer (VMD) drying. After mulberry leaves from the pre-drying method using VMD at 4,800 W were crushed and 50 mesh sieved. During drying with combination method, the drying time, the production yield and the production cost (mulberry leaf, electricity and equipment depreciation cost) were determined. The physical characteristics of mulberry leaf powder were determined: color value (L^* , a^* and b^*), bulk density, solubility, hygroscopic and angle of repose (modified from Jaya and Das, 2004). In addition, 1-deoxynojirimycin (DNJ), total chlorophyll, total phenolic compounds and antioxidant activities (using DPPH, ABTS and FRAP methods) were determined and discussed.

3. Results and Discussions

3.1 Physicochemical compositions and bioactive compounds of mulberry leaves

The different fresh mulberry leaf maturity levels had different in size and weight. Old leaves had the highest values of width, length and weight (16.44 ± 0.87 , 22.84 ± 1.61 and 5.98 ± 1.25 cm, respectively) (Table 1). It was found that the older of leaves had the darker of green color. When the chemical compositions of each maturity levels were analyzed, the results showed that young leaves had the highest amounts of protein and fat ($24.40 \% \pm 0.34$ and $1.21 \% \pm 0.01$, respectively). Mature and old leaves had the similar amounts of protein ($19.87 \% \pm 1.02$ and $19.98 \% \pm 0.41$, respectively). The Queen Sirikit Department of Sericulture (2558) reports of protein, fat, ash, crude fiber and carbohydrate of fresh mulberry leaf had 22.60, 4.57, 11.00, 10.36 and 42.25%, respectively. As is evident, this study has revealed lower amounts of fat and carbohydrate than that report. The different physicochemical of fresh mulberry leaf may result from different growing areas, seasons, and watering the plant. The mature leaves had the highest amounts of 1-Deoxynojirimycin (DNJ) (2.38 mg/g dried basis) (Table 2). DNJ content was contrast result to Wiroj (2009) report which there was the highest amount in young leaves (1.69 ± 0.70 mg/g). The old leaves had the highest amounts of total phenolic compound ($6.90 \pm 0.01 \mu\text{mol}$ of GAE/g dried basis) (Table 2). All maturity levels of mulberry leaves had the similar antioxidant activity (DPPH and ABTS assay), but mature leaves had the highest value of antioxidant activity from FRAP assay. So, in practical for commercial production, all leaves from each harvested branches of mulberry could be used as raw materials for mulberry leaf powder production. Consideration to bioactive compounds and antioxidant activities, all maturities of mulberry leaves had slightly different amounts. When all maturity levels of mulberry leaves were mixed and analyzed, it was shown that there were DNJ 1.58 mg/g (dried basis), phenolic compounds 3.45 mg/g (dried basis), antioxidant activities which were assayed using DPPH, ABTS and FRAP methods, there were 1.76 , $3.69 \mu\text{mol}$ of trolox/g dried basis and $20.10 \mu\text{mol}$ of Fe^{2+}/g (dried basis), respectively. The mixed leaves had $475.00 \text{ mg}/100\text{g}$ sample of total chlorophylls content. This amount was higher than alfalfa ($225 \text{ mg}/100\text{g}$ sample) which was used as raw materials for commercial chlorophyll production (colophyllmir, n.d.). It was able to indicate that mulberry leaf had 2 times higher amounts of chlorophylls than alfalfa. From this study, mixed maturity levels of mulberry leaf from Burirum 60 had high potential to use as raw material for chlorophyll production.

Table 1. Physicochemical composition of fresh mulberry leaves

Physical analysis	Young leaves	Mature leaves	Old leaves
Weight proportion (%)			
Width(cm)	5.28 ± 1.77	12.26 ± 1.61	16.44 ± 0.87
Length (cm)	7.50 ± 1.10	16.70 ± 2.00	22.84 ± 1.61
Weight(g)	0.56 ± 0.21	2.72 ± 0.41	5.98 ± 1.25
Color			
- L*	44.43 ± 4.22	48.66 ± 3.15	48.76 ± 0.56
- a*	-5.98 ± 4.52	-15.06 ± 3.18	-17.84 ± 0.72
- b*	27.41 ± 6.87	30.78 ± 4.29	30.93 ± 1.58
Chemical analysis			
Moisture content(%)	76.12 ± 3.14	76.12 ± 3.14	76.12 ± 3.14
Protein(%db)	24.40 ± 0.34	19.87 ± 1.02	19.98 ± 0.41
Fat (%db)	1.21 ± 0.01	0.47 ± 0.13	0.53 ± 0.11
Ash (%db)		8.57 ± 0.23	13.80 ± 0.80
Crude fiber (%db)	8.97 ± 0.36	9.87 ± 1.02	12.47 ± 1.98
Carbohydrate (%db)		55.95	53.39
			53.80

Table 2. Bioactive compounds of fresh mulberry leaf

Bioactive compounds	Young leaves	Mature leaves	Old leaves
1-Deoxynojirimycin (mg/g dried basis)	1.59	2.38	0.64
Total phenolic compound (μmol of GAE/g dried basis)	5.81 ^c ± 0.01	6.39 ^b ± 0.01	6.90 ^a ± 0.01
Antioxidant activities			
- DPPH ^{ns} (μmol of trolox/g dried basis)	1.19 ± 0.02	1.01 ± 0.04	0.92 ± 0.22
- ABTS ^{ns} (μmol of trolox/g dried basis)	10.71 ± 0.02	10.89 ± 0.18	10.91 ± 0.25
- FRAP (μmol of Fe ²⁺ /g dried basis)	32.83 ^b ± 0.02	40.16 ^a ± 0.01	24.06 ^c ± 0.01

Note: Different superscripts in the same row indicate statistically significant values ($P \leq 0.05$); ns mean values in the same row are not significantly different ($P > 0.05$)

3.2 Effect of heat pump dryer (HPD) compare with vacuum microwave dryer (VMD) on mulberry leaf qualities

When the fresh mulberry leaves were dried using heat pump dryer at 60 °C, it was showed that the maximum load capacity of fresh mulberry leaves was 3,500 g per batch.

VMD that used in this research the drying chamber had cylinder shape and rotating drying basket. It has 6 magnetron power each had the maximum 800 W. When fresh mulberry leaves were filled into drying basket of the VMD, it was found that the maximum filling weight was 700 g per batch. At 4,800 W magnetron power drying with 10 minutes drying time could provide mulberry leaves powder. If single stage drying using VMD, 4,800 W magnetron power was suitable for mulberry leaves drying which had only 10 minutes drying time (Table). This method could dry 700 g of fresh mulberry leaves and provide 141.61 g per batch. The electricity cost 10.71 ± 1.67 baht/kg of leaf powder. The analysis of bioactive compounds showed that there were phenolic compounds 5.87 mg/g (dried basis), antioxidant activities which were assayed using DPPH, ABTS and FRAP methods, there were 22.60, 11.54 μ mol of trolox/g dried basis and 46.94 μ mol of Fe²⁺/g (dried basis), respectively.

Table 3 Effect of HPD and VMD of drying on qualities and bioactive compounds in mulberry leaf powder

Qualities	Quality values	
	HPD	VMD
During drying		
-Initial mulberry leaf moisture content (%)	75.16 \pm 0.97	79.10 \pm 0.35
-Drying time (h)	12 \pm 0.00	0.16 \pm 0.00
-Yield (%)		20.23 \pm 2.55
-Final moisture content (%)	5.24 \pm 0.57	5.40 \pm 0.53
-Electricity cost (baht/kg of leaf powder)	151.2 \pm 3.89	10.71 \pm 1.67
Bioactive compounds		
-Total phenolic compounds (μ mol of GAE/g dried basis)	4.09 \pm 0.04	5.87 \pm 0.36
-Antioxidant activities		
- DPPH (μ mol of trolox/g dried basis)	11.58 \pm 0.01	22.60 \pm 3.47
- ABTS (μ mol of trolox/g dried basis)	13.26 \pm 0.89	11.54 \pm 1.70
- FRAP (μ mol of Fe ²⁺ /g dried basis)	56.77 \pm 0.02	46.94 \pm 8.58

3.3 Effect of combination method for mulberry leaf drying

Although drying in vacuum microwave dryer at 4,800 W magnetron power for 10 minutes maybe the suitable for mulberry leaf powder production, but from observation the drying basket can fill only 700 g per set if fill more than this even the drying basket is rotated but the leaves inside had too tight until unable to move. These effected to the leaves cannot get the uncertain microwave the leaves not equal dry some too much dry, some still fresh. The concept of this research is picked the withered leaves that passed the pre-dried from HPD and transfer to dry in VMD.

When the combination drying of HPD and VMD was studied. From the previous study, after primary dewatering of 3,500 g fresh mulberry leaves using heat pump dryer at 60 °C could provide 1,800 g of primary dewatered leaves. This amount of dewatered leaves was maximum amount to fill into drying basket of VMD. After drying using VMD at 4,800 W magnetron power, it was found that drying time was 20 minutes, production yield from fresh mulberry leaves was $27.24 \pm 4.21\%$. The final moisture content of mulberry leaves powder was $3.9 \pm 2.10\%$. The total drying time using combination method was $15 + 20 = 35$ minutes. Consideration to only electricity cost, this combination method was 11.55 ± 0.54 baht/kg of leaf powder. The finished mulberry leaf powder had $5.29 \mu\text{mol}$ of GAE/g dried basis of phenolic compounds and $786.00 \text{ mg}/100\text{g}$ sample of total chlorophylls. For antioxidant activities which were assayed using DPPH, ABTS and FRAP methods, there were $8.10 \mu\text{mol}$ of trolox/g dried basis, $16.23 \mu\text{mol}$ of trolox/g dried basis and $54.59 \mu\text{mol}$ of Fe^{2+} /g dried basis, respectively.

Table 5. Effect of HPD and VMD combination on qualities and bioactive compounds in mulberry leaf powder

Quality characteristics	Quality values
During primary dewatering	
-Initial mulberry leaf moisture content (%)	78.4 ± 3.61
-Weight of fresh mulberry leaf (kg)	3.50
-Weight of mulberry leaf after pre-dewatering (kg)	1.80
-Drying time of primary dewatering (min)	15.00
-Final moisture content (%)	38.5 ± 1.05
During final dewatering	
-Initial mulberry leaf moisture content after primary dewatering (%)	38.5 ± 1.02
-Weight of mulberry leaf (kg)	1.50
-Weight of dry mulberry leaf (kg)	0.50
-Drying time (min)	20.00
-Final moisture content (%)	3.9 ± 2.10
-Final yield from fresh mulberry leaf (%)	27.2 ± 4.21
-Electricity cost (baht/kg leaf powder)	11.55 ± 0.54
Physical qualities	
-Color of leaf powder	
- L*	59.4 ± 0.67
- a *	-17.6 ± 0.24
- b *	25.7 ± 0.42
-Bulk density (g/100ml)	71.8 ± 0.83
-Solubility (%)	18.8 ± 1.81
-Hygroscopicat RH = 75% (%)	0.2 ± 0.44

-Angle of repose (°)	47.5 ± 0.31
Bioactive compounds	
- 1-Deoxynojirimycin (mg/g dried basis)	3.23
-Total phenolic compounds(μmol of GAE/g dried basis)	5.29 ± 0.62
-Total chlorophylls (mg/100 g sample)	786.00
-Antioxidant activities	
- DPPH (μmol of trolox/g dried basis)	8.1 ± 0.05
- ABTS (μmol of trolox/g dried basis)	16.2 ± 0.56
- FRAP (μmol of Fe ²⁺ /g dried basis)	54.6 ± 3.47

4. Conclusions

All of maturities had slightly different on bioactive compounds and antioxidant activities. In practical, it can be used mixed mulberry leaf for mulberry leaf powder production. Primary dewatering preparation of mulberry leaf to 50% moisture content of fresh leaves could be done by drying in heat pump dryer at 60 °C for 15 minutes. For final dewatering step to about 4% moisture content, it could be done by drying in vacuum microwave dryer at 4,800 W magnetron power for 20 minutes. This combination method provided 27.24% of production yield from fresh mulberry leaf. Its electricity cost was 11.55 baht/kg of leaf powder. The mulberry leaf powder had 5.29 μmol GAE/g (dried basis) of phenolic compounds, 786.00 mg/100g sample of total chlorophylls and ...mg/g (dried basis) of 1-Deoxynojirimycin. For antioxidant activities which were assayed using DPPH, ABTS and FRAP methods, there were 8.10 μmol of trolox/g (dried basis), 16.23 μmol of trolox/g (dried basis) and 54.59 μmol of Fe²⁺/g (dried basis), respectively. From this study, this mulberry leaf powder product had high potential to be supplementary food which could be directly or encapsulated consumed.

Acknowledgements

The authors are grateful for the financial support to this research from the Haze Free Thailand (Chiang Mai University). Special thanks to The Queen Sirikit Department of Sericulture (Chiang Mai, Thailand) for supporting the entire mulberry leaves for this research.

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