



## Physicochemical Characteristics of Essential Oil of Black Pepper (*Piper nigrum*) Cultivated in Chittagong, Bangladesh

S. Morshed<sup>1\*</sup>, M.D. Hossain<sup>2</sup>, M. Ahmad<sup>1</sup>, M. Junayed<sup>3</sup>

1. Department of Applied Chemistry and Chemical Technology, Chittagong Veterinary and Animal Sciences University, Khulshi, Chittagong-4225, Bangladesh

2. Bangladesh Standards and Testing Institutions (BSTI), 116/A, Tejgaon I/A, Dhaka-1208, Bangladesh

3. Forest Protection Division, Bangladesh Forest Research Institute, Sholashahar, Chittagong-4211, Bangladesh

### HIGHLIGHTS

- Major components of black pepper Essential Oil (EO) were caryophyllene (19.12%), limonene (9.74%), and camphene (8.44%).
- Refractive index of black pepper EO was 0.8590.
- Having desirable physicochemical characteristics, black pepper EO cultivated in Bangladesh could be applied in food industry.

### Article type

Original article

### Keywords

Spices  
Oils, Volatile  
Food Analysis

### Article history

Received: 22 May 2017

Revised: 3 Jul 2017

Accepted: 27 Jul 2017

### Acronyms and abbreviations

EO=Essential Oil  
GC/MS=Gas Chromatog-  
raphy/Mass Spectrometry

### ABSTRACT

**Background:** Black pepper (*Piper nigrum*) is mainly cultivated in tropical areas such as South India. It is known as a popular spice in all over the world. Although some studies have been carried out to determine the ingredients of black pepper Essential Oil (EO) cultivated in various regions of the world, but there is little data about some physicochemical characteristics of EO of black pepper cultivated in Chittagong, Bangladesh.

**Methods:** Black peppers samples were collected from the local market of Chittagong city. Extraction of black pepper EO was carried out using Clevenger apparatus. Chemical compounds of EO obtained from the black pepper were determined by analytical gas chromatography/mass spectrometry. Some physicochemical characteristics of extracted EO were determined according to the standard methods. All experiments were carried out in triplicate.

**Results:** The major components of black pepper EO were caryophyllene (19.12%), limonene (9.74%), and camphene (8.44%). The contents of moisture, dry matter, protein, fatty oil, ash, carbohydrate, and crude fiber in black pepper samples were determined as 2.20, 96.12, 12.66, 14.41, 12.49, 42.56, and 5.55%, respectively. Also, food energy value was measured as 349.66±0.44 g/calories. Refractive index, specific gravity, as well as optical rotation of EO samples were 0.8590, 1.476 at 30 °C, and -10.300, respectively.

**Conclusion:** The EO of black pepper cultivated in Chittagong, Bangladesh revealed desirable physicochemical characteristics and could be applied in food industry.

### Introduction

Black pepper (*Piper nigrum*) is mainly cultivated in tropical areas such as South India. It is known as a popular spice in all over the world (Agbor et al., 2006). Such plants have a variety of compounds that comprise medic-

inal and nutritive values and food industries applicants. Also, the herbal spices have some antimicrobial and flavoring properties (Souza et al., 2005). In this regard, many researchers have reported variability in type and level of components of herbal spices depending on their geographical origin (Edeoga et al., 2003; Parthasarathy et al., 2008).

\* Corresponding author. ✉ sourabh\_acct@yahoo.com

**To cite:** Morshed S., Hossain M.D., Ahmad M., Junayed M. (2017). Physicochemical characteristics of essential oil of black pepper (*Piper nigrum*) cultivated in Chittagong, Bangladesh. *Journal of Food Quality and Hazards Control*. 4: 66-69.

Essential Oils (EOs) are recognized as aromatic, odoriferous, and volatile oily liquids extracted from various parts of herbs by some laboratorial methods such as steam distillation, expression, etc. It has been approved that some plants EOs have antioxidant, antimicrobial, and also anticancer properties (Amorati et al., 2013; Bakkali et al., 2008; Calo et al., 2015; Nazzaro et al., 2013; Seow et al., 2014; Teixeira et al., 2013; Tongnuanchan and Benjakul, 2014). Although some investigations have been carried out to determine ingredients and physicochemical properties of black pepper EO cultivated in various regions of the world, but there is little data about some physicochemical characteristics of EO of black pepper (*P. nigrum*) cultivated in Chittagong, Bangladesh.

## Materials and methods

### Preparation of black peppers

Black peppers were collected from the local market of Chittagong city (Khatunganj). The samples were washed clearly by water to remove dust materials and then dried. Then, the dried black peppers were ground by mortar grinder (Fritsch, Germany) for one h. The final sieved powder was obtained with approximate average particle diameters of 0.25 and 0.50 mm.

### Extraction of EO

Extraction of black pepper EO was carried out using Clevenger apparatus (Germany) by steam distillation over a three h period. After preparation of the samples for Gas Chromatography/Mass Spectrometry (GC/MS) analysis, chemical compounds of EO of the black pepper were determined by analytical GC (Shimadzu, Japan) according to the standard method (Shavisi et al., 2017).

### Physicochemical analysis

Some physicochemical characteristics of extracted EO were determined according to the standard methods (British Pharmacopoeia Commission, 2011). Before proximate analysis, sample was allowed for sun drying and then the percentages of moisture, ash, nitrogen, protein, carbohydrate, crude fiber, in addition to total energy were measured according to the routine procedures (Aziz et al., 2012; Hossain et al., 2015; Saleh-E-In and Roy, 2007). All experiments were carried out in triplicate.

## Results

The major components of black pepper EO were caryophyllene (19.12%), limonene (9.74%), as well as

camphene (8.44%) as indicated in Table 1. The contents of moisture, dry matter, protein, fatty oil, ash, carbohydrate, and crude fiber in black pepper samples were determined as 2.20, 96.12, 12.66, 14.41, 12.49, 42.56, and 5.55%, respectively. Also, food energy value was measured as  $349.66 \pm 0.44$  g/calories. Refractive index, specific gravity, and optical rotation of EO samples were 0.8590, 1.476 at 30 °C, and  $-10.30^{\circ}$ , respectively. Chemical compositions of black pepper EO are shown in Table 2.

## Discussion

The results of physical and chemical analyses obtained in the present study were in accordance with the similar previous research carried out by Aziz et al. (2012). However, the acid value determined in samples of this study indicated lower proportion of free fatty acid content of black pepper (0.37) than those in edible oils like soybean oil (0.38-0.54), mustard oil (3.65-4.56), as well as palm oil (0.17-1.06) (Nollet, 2004). Considering high phenolic content found in the pepper samples, it seems that EO of black pepper is appropriate free radical scavenger for human health. The proximate composition of black pepper samples of the current investigation was in close agreement with Nwofia et al. (2013) who analyzed nutritional composition of *P. nigrum* (L.) accessions from Nigeria. Also, the black pepper samples had fairly high food energy value which might be due to the high lipid and carbohydrate content which showed some similarity with findings of Hossain et al. (2015) and Kimbonguila et al. (2010). However, little dissimilarity in the case of carbohydrate and protein levels was also seen that may be related to some environmental factors like temperature, rate of photosynthesis, etc. (Parthasarathy et al., 2008).

The major components identified by GC/MS analysis of EO samples of this study were caryophyllene (a sesquiterpene), limonene as well as camphene (both monoterpenes) which were close to the findings of Aziz et al. (2012). However, Jirovetz et al. (2002) had observed that the main compounds from EO of dried fruits of black pepper from Cameroon were germacrene D (11.01%), limonene (10.26%),  $\beta$ -pinene (10.02%),  $\alpha$ -phellandrene (8.56%),  $\beta$ -caryophyllene (7.29%),  $\alpha$ -pinene (6.40%), as well as *cis*- $\beta$ -ocimene (3.19%). These differences are probably due to various geographic and ecological conditions. Other important components were  $\beta$ -pinene (8.0%), 3-carene (7.08%),  $\alpha$ -pinene (6.32%), sabinene (2.98%), trifluoromethanesulfonyl fluoride (2.14%), and also caryophyllene oxide (1.78%) which are often responsible for different flavor and also aroma functions.

**Table 1:** GC/MS analysis of EO of black pepper cultivated in Chittagong, Bangladesh

No.	Compound	Retention time	%	Formula	Molecular weight
1	$\alpha$ -phellandrene	2.317	0.40	C <sub>10</sub> H <sub>16</sub>	136
2	Camphene	2.693	0.13	C <sub>10</sub> H <sub>16</sub>	136
3	Sabinene	2.752	2.98	C <sub>10</sub> H <sub>16</sub>	136
4	$\beta$ -pinene	2.869	8.00	C <sub>10</sub> H <sub>16</sub>	136
5	$\alpha$ -pinene	2.974	6.32	C <sub>10</sub> H <sub>16</sub>	136
6	3-Carene	3.181	7.08	C <sub>10</sub> H <sub>16</sub>	136
7	Trifluoromethanesulfonyl fluoride	3.253	2.14	CF <sub>3</sub> SO <sub>2</sub> F	152
8	(+)-Camphene	3.308	8.44	C <sub>10</sub> H <sub>16</sub>	136
9	0-Cymene	3.376	0.63	C <sub>10</sub> H <sub>14</sub>	134
10	Limonene	3.588	9.74	C <sub>10</sub> H <sub>16</sub>	136
11	$\beta$ -phellandrene	3.623	0.94	C <sub>10</sub> H <sub>16</sub>	136
12	Gamma-terpinene	3.998	0.11	C <sub>10</sub> H <sub>16</sub>	136
13	Terpinolene	4.414	0.30	C <sub>10</sub> H <sub>16</sub>	136
14	Terpinolene	4.500	0.36	C <sub>10</sub> H <sub>16</sub>	136
15	Linalol	4.584	0.15	C <sub>10</sub> H <sub>18</sub> O	154
16	4-terpineol	6.364	0.15	C <sub>10</sub> H <sub>18</sub>	154
17	Delta-Elementene	9.209	0.65	C <sub>15</sub> H <sub>24</sub>	204
18	Copaene	9.939	0.70	C <sub>15</sub> H <sub>24</sub>	204
19	$\beta$ -Elementene	10.142	1.15	C <sub>15</sub> H <sub>24</sub>	204
20	$\alpha$ -Bergamotene	10.540	0.62	C <sub>15</sub> H <sub>24</sub>	204
21	Caryophyllene	10.755	19.12	C <sub>15</sub> H <sub>24</sub>	204
22	$\alpha$ -Caryophyllene	11.265	1.83	C <sub>15</sub> H <sub>24</sub>	204
23	$\alpha$ -Curcumene	11.566	0.41	C <sub>15</sub> H <sub>22</sub>	202
24	Cedrene	11.640	0.64	C <sub>15</sub> H <sub>24</sub>	204
25	$\beta$ -Bisabolene	11.967	1.65	C <sub>15</sub> H <sub>24</sub>	204
26	(+)-delta-Cadinene	12.145	0.32	C <sub>15</sub> H <sub>24</sub>	204
27	Caryophyllene oxide	13.162	1.78	C <sub>15</sub> H <sub>24</sub> O	220
28	Spathulenol	13.729	0.44	C <sub>15</sub> H <sub>24</sub> O	220
29	$\alpha$ -Bisabolol	14.498	0.41	C <sub>15</sub> H <sub>26</sub> O	222
30	2-Undecanone	12.643	0.10	C <sub>11</sub> H <sub>22</sub> O	170

**Table 2:** Chemical compositions of EO of black pepper cultivated in Chittagong, Bangladesh

Characteristics	Results
Acid value	0.37±0.55
Aldehyde value	38.83±0.76
Alcohol content	62.87±0.41%
Ester number after acetylation	195.56±0.66
Phenol content	78±0.12%

## Conclusion

The EO of black pepper cultivated in Chittagong city, Bangladesh revealed desirable physicochemical characteristics and could be applied in food industry.

## Conflicts of interest

The authors declare no conflicts of interest.

## Acknowledgments

This study was financially supported by Chittagong Veterinary and Animal Sciences University. This research was ethically approved by the local institutional review board. We thank Mr. Delowar Hossain (Field Officer, Bangladesh Standards and Testing Institutions, 116/A, Tejgaon I/A, Dhaka-1208, Bangladesh) for his valuable help during the project. We are also grateful to Mr. Monsur Ahmad (Lecturer, Department of Applied Chemistry and Chemical Technology, of Chittagong Veterinary and Animal Sciences University, Chittagong) for his active cooperation.

## References

- Agbor G.A., Vinsonb J.A., Obenc J.E., Ngogangd J.Y. (2006). Comparative analysis of the *in vitro* antioxidant activity of white and black pepper. *Nutrition Research*. 26: 659-663.
- Amorati R., Foti M.C., Valgimigli L. (2013). Antioxidant activity of essential oils. *Journal of Agricultural and Food Chemistry*. 61: 10835-10847.
- Aziz S., Naher S., Abukawsar M., Roy S.K. (2012). Comparative studies on physicochemical properties and GC-MS analysis of essential oil of the two varieties of the black pepper (*Piper nigrum* Linn.). *International Journal of Pharmaceutical and Phytopharmacological Research*. 2: 67-70.
- Bakkali F., Averbeck S., Averbeck D., Idaomar M. (2008). Biological effects of essential oils—a review. *Food and Chemical Toxicology*. 46: 446-475.
- British Pharmacopoeia Commission (2011). British Pharmacopoeia. London, UK.
- Calo J.R., Crandall P.G., O'Bryan C.A., Ricke S.C. (2015). Essential oils as antimicrobials in food systems—a review. *Food Control*. 54: 111-119.
- Edeoga H.O., Okwu D.E., Mbaebie B.O. (2003). Minerals and nutritive value of some Nigerian medicinal plants. *Journal of Medicinal and Aromatic Plant Science*. 25: 1010-1015.
- Hossain M.D., Paul B.K., Roy S.K., Saha G.C., Begum F., Huq D. (2015). Studies on fatty acids composition and some valuable nutrients of *Piper nigrum* Linn. (Gol Morich). *Dhaka University Journal of Science*. 62: 65-68.
- Jirovetz L., Buchbauer G., Ngassoum M.B., Geissler M. (2002). Aroma compound analysis of *Piper nigrum* and *Piper guineense* essential oils from Cameroon using solid-phase microextraction–gas chromatography, solid-phase microextraction–gas chromatography–mass spectrometry and olfactometry. *Journal of Chromatography A*. 976: 265-275.
- Kimbonguila A., Nzikou J.M., Matos L., Loumouamou B., Ndangui C.B., Pambou-Tobi N.P.G., Desobry S. (2010). Proximate composition and physicochemical properties on the seeds and oil of *Annona muricata* grown in Congo Brazzaville. *Research Journal of Environmental and Earth Science*. 2: 13-18.
- Nazzaro F., Fratianni F., De Martino L., Coppola R., De Feo V. (2013). Effect of essential oils on pathogenic bacteria. *Pharmaceuticals*. 6: 1451-1474.
- Nollet L.M.L. (2004). Hand book of food analysis, physical characterization and nutrient analysis. Marcel Dekker Inc., New York.
- Nwofia G.E., Kelechukwu C., Blessing K. (2013). Nutritional composition of some *Piper nigrum* (L.) accessions from Nigeria. *International Journal of Medicinal and Aromatic Plants*. 3: 247-254.
- Parthasarathy V.A., Chempakam B., Zachariah T.J. (2008). Chemistry of spices. CAB International, London.
- Saleh-E-In M.M., Roy S.K. (2007). Studies on fatty acid composition and proximate analyses of *Anethum sowa* L. (dill) seed. *Bangladesh Journal of Scientific and Industrial Research*. 42: 455-464.
- Seow Y.X., Yeo C.R., Chung H.L., Yuk H.G. (2014). Plant essential oils as active antimicrobial agents. *Critical Reviews in Food Science and Nutrition*. 54: 625-644.
- Shavisi N., Akhondzadeh Basti A., Khanjari A., Misaghi A., Shahbazi Y., Hajjar Bargh A., Vanaki E. (2017). *In vitro* antibacterial activity of poly(lactic acid) film incorporated with ethanolic propolis extract and *Ziziphora clinopodioides* essential oil. *Journal of Food Quality and Hazards Control*. 4: 3-8.
- Souza E.L.D., Lima E.O., Trajano V.N., Filho J.B. (2005). Antimicrobial effectiveness of spices: an approach for use in food conservation systems. *Brazilian Archives of Biology and Technology*. 48: 549-558.
- Teixeira B., Marques A., Ramos C., Neng N.R., Nogueira J.M., Saraiva J.A., Nunes M.L. (2013). Chemical composition and antibacterial and antioxidant properties of commercial essential oils. *Industrial Crops and Products*. 43: 587-595.
- Tongnuanchan P., Benjakul S. (2014). Essential oils: extraction, bioactivities, and their uses for food preservation. *Journal of Food Science*. DOI: 10.1111/1750-3841.12492.