

Physicochemical properties of ambient and cryoground black pepper

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Abstract

The physicochemical characteristics (moisture, particle size, bulk density, true density, porosity, angle of repose, coefficient of friction, color, oil, oleoresin and piperine) of ambient and cryogenically ground black pepper determined on the basis of particle size (71.04 - 491.90 μm) at both conditions. The bulk and true densities and angle of repose decreased with the increase of particle size of both conditions and varied from 532.9-419.2 kg/m³, 615.9-563.4 kg/m³ and 41.8-30.9°, respectively. The porosity has increased from 24.2 to 25.6 percent with the increase of grades. Oil, oleoresin and piperine has significantly more retained in cryoground samples and varied from 1.2-1.6%, 6.5-11.9% and 1.8-4.1%, respectively. *L* values increased for ambient conditions. Values for *a* and *b* were decreased with increase of grades at both conditions and varied from 15.96-44.15, 4.62-3.59 and 19.29-12.93, respectively. The results showed that moisture content had no significant effects on grades but other properties had highly significant.

Keywords: Black pepper, Cryogenic grinding, Physical properties, Physicochemical properties.

Introduction

The black pepper is one of the most important spices in India. Black pepper is the most pungent and flavorful of all types of peppers and it is available as whole or cracked peppercorns or ground into powder. There are several varieties of black pepper seeds grown throughout different parts of India and compositional variations (Balasubramanian *et al.*, 2016). The physical and chemical properties are for value addition and storage conditions also play important role (Singh and Goswami 2000). It is also a good source of medicinal values. In India, the domestic consumption of black pepper was 52000 tons in 2002 which rose to 58,000 tons in 2003. Cryogenic spice grinding has many advantages over traditional grinding procedures (Balasubramanian *et al.*, 2012). Since physicochemical property of any product determines its quality, Sarikaya *et al.* (2001) determined some physicochemical properties of fine alumina powder on the basis of electron microscopy. Sablani *et al.* (2008) determined

physicochemical properties of date powder using new techniques such as water activity, bulk density, color, hygroscopicity and glass transition temperatures and the date paste dried with 1.0 kg of maltodextrin 1.0 kg⁻¹ of date paste produced non sticky and free flowing powder. In case of wild variety, the total phenolic contents were found to be 32.32 g/100 g of gallic acid equivalent (dwb), whereas Chakiya variety had 24.50 g/100 g of gallic acid equivalent (dwb). Miyamoto *et al.*, 2009 also studied the effect of heating conditions on physicochemical properties of skim milk powder during production and found turbidity increased with an increase in integrated caloric content. Physical properties are useful in post-harvest unit operations for the design of cleaning, grading, sorting, transportation, handling, aeration, sizing, storing, size reduction, packaging and other processing equipment (Sahay and Singh, 2001). Therefore, present work was undertaken to determine the physical and chemical characteristics of ambient as well as cryoground ground black pepper and to see the effect of different grades on these properties.

Material and Methods

Raw materials and preparation of powder

Black pepper seeds were procured from local market, Ludhiana, Punjab (India). Before the samples were prepared for the experiment, the seeds were cleaned manually and broken, foreign matter, split, deformed and immature seeds were discarded. The initial moisture content of seed was determined (Ranganna, 1986). The initial moisture content of the black pepper was found to be 8.3 % on dry basis (Balasubramanian *et al.*, 2013a). Initially the seeds were stored at ambient condition (25°C) for 2 to 3 weeks. For experimentation, about 10 kg of black pepper was taken for grinding and grounded in the pin mill type grinder (100 UPZ, Hosokawa Alpine, Germany) at ambient condition and cryogenic conditions (<-50°C) using liquid nitrogen (LN₂) at grinder speed of 2000 rpm and 1 kg h⁻¹ feed rate. Thus obtained ground black pepper samples of both conditions were used for determination of physicochemical properties of black pepper.

Preparation of grades and determination of properties

For measurement of the physicochemical properties, powder was graded into three different sizes based on its particle size using BSS sieves viz. 50, 85 & pan. The moisture content was determined of all grades of powders using standard hot air oven method at 70°C until the constant weight achieved and replicated in triplicate. (AOAC, 2000). The particle size of graded samples were determined as 85, 284, 492 µm for ambient and 71, 211, 322 µm for cryogenic grinding conditions using particle size analyzer (LA 950, Horiba, Japan). The average bulk density was determined using the standard method by filling a measuring cylinder of 100 ml with the powder from a constant height (Singh and Goswami, 1996) and it is the ratio of the mass.

The average true density is the ratio of the mass of the seed to its pure volume and determined using gas (nitrogen) pycnometer (model 2: Hymipyc and make IQI, USA). Before starting of the experiment, the pycnometer was calibrated using calibration kit and standard volume of steel ball 1.0725 cc. After calibration, the triplicate weighed samples were put in sample chamber for determination of true density and volume in plot the graph between time and pressure and the results were printout. Chang (1988) has determined the density and porosity of grain kernels using gas pycnometer. In this study, the nitrogen gas filled pycnometer used and the maximum pressure was 300 kPa. Pycnometer carrier

gas is delivered to the sample chamber by opening the gas valve and controlling the voltage to achieve desired pressure value. Upon establishing a steady pressure reading from transducer and assuming prior establishing of ambient pressure in the reference chamber, the second proportional valve is opened in a controlling way to allow for continuous transfer of gas from sample chamber to reference chamber. The exhaust valve and the vacuum valve are closed during this operation. The established pressure value after depressurization is read for subsequent calculations. The second proportional valve is also used in similar way to either establish ambient pressure in the sample chamber, or when connecting vacuum port in the sample chamber.

Porosity is the fraction of the space in the bulk seeds which is not occupied by the seeds (Thompson and Issacs, 1967, Balasubramanian, 2013b). The porosity of bulk seed was calculated from the values of true density and bulk density using the relationship given by Mohsenin (1986) as follows:

... (1) where, θ is the porosity, %; ρ_t is the true density, kg/m³ and ρ_b is the bulk density, kg/m³. A tapering hopper made of iron with the top and bottom having a dimension of 250mm×250mm×250mm and 20mm×20mm hole in the bottom, respectively. At 210mm from the bottom of hopper, a circular disc of 100mm diameter was used so that enough gap was left between the hopper wall and the disc which allows the seed to flow through during the test. A horizontal sliding gate was provided right below the disc for sudden release of the seeds during the test. A similar device was used by Sahoo and Srivastava (2002) for okra (*Abelmoschus esculentus*) seed. The angle of repose was calculated from the measurement of the height of the heap of seeds on a circular plate as given in equation

$$\theta = \arctan\left(\frac{H}{D}\right)$$

where, H is the height of the cone in cm and D is the diameter of iron plate in cm. The static coefficient of friction (μ_s) of coriander seeds were determined (Ozturk and Esen 2008) with respect to four surfaces: plywood, galvanized iron sheet, mild steel sheet and aluminum. These materials are mostly used for construction of storage structures. A laboratory setup was used to calculate the static coefficient of friction. During the

experiment a leveled rectangular box was filled of the seeds at desired moisture content and moved on the frictional surface with the help of rope and pulley. The frictional force and normal strength was noted and the same quantity of seed was used on the other frictional surfaces of the same moisture content. It was calculated by using the equation,

$$\mu_s = \frac{F}{N_f}$$

Where, F is the measured force in N and N_f is the normal strength of the samples in N.L, a and b parameters were determined using a Hunter color meter (model Miniscan Xe plus, USA) in the laser reflectance mode where, L is the lightness or darkness (black, $L = 0$; white, $L = 100$), $+a$ is redness, $-a$ is greenness, $+b$ is yellowness, and $-b$ is blueness. The instrument was calibrated with white standard calibration plate provided by the manufacturer ($L = 97.59$, $a = -5.06$, $b = 6.91$). Black pepper powders were spread to 5 mm thick over the white paper, and the tip of the measuring head was pointed on the samples for measurement. Three to five measurements were recorded for each sample and reported average data.

Essential oil, oleoresin and piperine were estimated by the ASTA method from ground samples. Essential oil was extracted by hydro distillation using Clevenger trap for lighter than water type from powdered sample. The oil collected is computed as percentage oil (volume/dry weight in 100 g). Oleoresin was extracted from the powdered sample by cold percolation technique and the viscous mass obtained was computed as percentage oleoresin (weight/dry weight in 100 g). Piperine from the powdered sample was extracted by refluxing in alcohol and estimated by HPLC and the percentage was computed based on authentic standard. The physical and chemical properties of black pepper powder were determined and the experiments were conducted in triplicate at different grades.

Data Analysis

Statistical analysis black pepper powder was carried out using Ag Res, version 7.01 (Pascal International Software solutions, USA) and statistical procedures described by Gomez and Gomez (1984) to examine the effect of grades ($p > 0.05$) on physical and chemical characteristics.

Results and Discussion

Physical characteristics

The physical properties of black pepper (*Piper nigrum*)

powder with different grades at ambient and cryoground conditions are given in Tables 1(a & b). The moisture content black pepper powder ranged from 5.31 to 7.39 % d.b. at both ground conditions and different particle size. The particle size varied from 84.9 to 491.9 μm at ambient ground samples and at cryo ground conditions, the particle size varied from 71 to 322.5 μm at three different grades. The particle size of ambient ground has larger than the cryoground samples due to injection of liquid nitrogen or cold grinding process. Singh and Goswami (1999) reported that the temperature increased from -160 to -70°C for 12 number rotor ribs, the volume mean diameter of cumin powder increased from 129 to 164 μm . Thus, the product temperature rise from 80-85°C at ambient conditions resulted that particle size increased (Goswami and Singh 2003). There was significant difference ($p < 0.05$) in physical properties between grades and grinding conditions. The bulk and true densities of black pepper powder decreased significantly of both conditions at different particle sizes and the bulk density varied in the range of 465.9 to 419.2 and 523.8 to 490.2 at ambient and cryo conditions and the true density in the range from 614.4 to 563.4 and 615.8 to 588.5 kg/m³, respectively at all conditions. The bulk and true densities are both higher of cryo conditions than the ambient. This might be resulted that the particle size more smaller during cryo grinding and the densities would be more. The porosity of both conditions also increased with the increase in grades and varied from 24.2 to 25.6%. This might be due to the particle size courser of higher grade. Similar results were reported by Singh and Goswami (1999) for cumin seeds. Angle of repose decreased from 33.9 to 30.9° at ambient conditions and 41.8 to 31.8° at cryo conditions of different grades.

Color measurement

The color of black pepper powder was dark red during cryoground conditions whereas an ambient conditions more changes in L , a and b . Therefore, the colour profile of these conditions was expected to differ with change grinding conditions using liquid nitrogen. The L , a and b values varied from 15.92 to 36.50, 4.36 to 4.19 and 19.05 to 12.93, respectively during ambient conditions. L , a and b values of cryo conditions varied from 44.15 to 30.33, 4.62 to 3.59 and 19.29 to 13.98, respectively at different grades. The color value were retains during cryo conditions due to cold grinding. The coefficient of friction of black pepper powders were determined at plywood, mild steel and galvanized iron sheets. The coefficient of

friction found higher at plywood of both conditions due to more rubies surface. The coefficient of friction at plywood, mild steel and galvanized iron sheets varied from 0.896 to 0.776, 0.610 to 0.836 and 0.648 to 0.687, respectively at ambient conditions. During cryo conditions, the coefficient of friction varied from 0.734 to 0.588, 0.734 to 0.504 and 0.584 to 0.504, respectively at plywood, mild steel and galvanized iron sheets of all grades. Singh and Goswami (1999) also determined the coefficient of friction at these surfaces of cumin seeds at different moisture content and found plywood had highest coefficient of friction.

Bio chemical properties

The bio chemical characteristics of black pepper powder at ambient and cryo conditions were determined using standard ASTA 1968 method and found from the results the essential oil has more retention at cryo conditions due to cold grinding. Singh and Goswami, 2000 studied the cryogenic grinding of cumin seeds and reported that 50% retains the volatile oil. The oil, oleoresin and piperine at ambient conditions varied from 1.2%, 6.6 to 7.1% and 1.8 to 2.2%, respectively at different grades. During cryo conditions, the oil, oleoresin and piperine varied from 1.2 to 1.6%, 6.5 to 11.9% and 1.9 to 4.1%, respectively. From the tables 1a and b found that the biochemical properties of black pepper powders had maximum oil retained at grade III at cryo conditions. This might be due to the grade III has more fine than other grades and found to be

better result. Zachariah et al. (2010) similar studied the chemical analysis of black pepper leaf and seeds and found ? caryophyllene higher in berries as compared to leaf oil.

Conclusions

This study examined the effect of physico chemical properties of ambient and cryo ground black pepper at different grades. The particle size has more fine during cryogenic grinding of black pepper. The bulk and true densities were decreased significantly as increases the grades. The porosity has increased with the increase in grades. The angle of repose and coefficient of frictions found decreased with increase in grades. The coefficient of friction had maximum at plywood at ambient and cryo conditions. The color value has more dark on cryo samples and brightness has also more during cryo conditions. The percentage values of oil, oleoresin and piperine found maximum during cryogenic grinding of samples. The oleoresin percentage had maximum 11.9% of grade III at cryo conditions. The bulk and true densities were decreased significantly as increases the grades.

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Table 1a. Physicochemical properties ambient ground black pepper

Grades	Moisture content (%d.b.)	Particle size ()	Bulk density (kg/m ³)	True density (kg/m ³)	Porosity (%)	Angle of repose (°)	Color			Coefficient of Friction			Biochemical		
							L	a	b	plywood Sheet	MS Sheet	GI (%)	Oil (%)	Oleoresin (%)	Piperine (%)
1.	7.39 ^c	84.9 ^a	465.87 ^c	614.4 ^c	24.2 ^b	33.9 ^a	15.92 ^c	4.36 ^a	19.05 ^a	0.896 ^a	0.610 ^c	0.648 ^a	1.2 ^a	6.7 ^b	2.0 ^b
2.	7.21 ^b	283.9 ^b	428.09 ^b	572.0 ^b	25.2 ^a	30.9 ^c	37.01 ^a	4.38 ^a	14.23 ^b	0.817 ^b	0.761 ^b	0.687 ^b	1.2 ^a	6.6 ^b	1.8 ^c
3.	7.18 ^a	491.9 ^c	419.18 ^a	563.4 ^a	25.6 ^a	32.8 ^b	36.50 ^b	4.19 ^b	12.93 ^c	0.776 ^c	0.836 ^a	0.677 ^b	1.2 ^a	7.1 ^a	2.2 ^a

Means in columns with different superscripts are significantly different at p>0.05

Table 1b.Physicochemical properties cryogenic ground black pepper

Grades	Moisture content (%d.b.)	Particle size ()	Bulk density (kg/m ³)	True density (kg/m ³)	Porosity (%)	Angle of repose (°)	Color			Coefficient of Friction			Biochemical		
							L	a	b	plywood Sheet	MS Sheet	GI (%)	Oil (%)	Oleoresin (%)	Piperine (%)
1.	6.71 ^a	71.0 ^c	523.81 ^a	615.8 ^a	24.2 ^b	41.8 ^a	44.15 ^a	4.62 ^a	19.29 ^a	0.734 ^b	0.734 ^a	0.584 ^a	1.2 ^a	7.8 ^b	2.2 ^b
2.	6.20 ^b	211.2 ^b	507.69 ^b	589.7 ^b	25.2 ^a	31.8 ^c	30.33 ^c	3.59 ^c	13.98 ^c	0.785 ^a	0.664 ^b	0.513 ^b	1.6 ^a	6.5 ^c	1.9 ^c
3.	5.31 ^c	322.5 ^a	490.23 ^c	588.5 ^c	25.6 ^a	36.7 ^b	32.97 ^b	4.32 ^b	14.26 ^b	0.588 ^c	0.504 ^c	0.504 ^b	1.6 ^a	11.9 ^a	4.1 ^a

Means in columns with different superscripts are significantly different at p>0.05

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