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An experimental study on cryogenic spice grinding system for black pepper grinding

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Abstract

In this paper, an experimental study on cryogenic spice grinding system for black pepper grinding has been presented. Black pepper was ground in developed cryogenic spice grinding system at varying temperatures (10°C and -50°C) and varying conveyor screw speeds (5, 10 and 15 rpm) in its pin mill (6500 rpm). Average particle size increased with increasing conveyor screw speed irrespective of temperature. A higher particle size was obtained at 10°C irrespective of conveyor screw speed in comparison to that obtained at -50°C. A more bright and light colour was obtained at -50°C in comparison to that at 10°C. The variation of *a, b,* chroma value, hue angle and browning index were significant ($p \leq 0.05$) with varying speed at -50°C and that of non-significant with varying speed at 10°C. The maximum values of piperine (2.65 %), total phenol-alcohol extract (1.00 %) and total phenol-water extract (0.87 %) were obtained at -50°C and 5 rpm. The maximum values of DPPH activity-alcohol extract (23.85 %) and DPPH activity-water extract (22.66 %) were obtained at -50°C and 5 rpm whereas minimum values of DPPH activity-alcohol extract (19.40 %) and DPPH activity -water extract (18.89 %) were obtained at 10°C and 15 rpm. It was observed that developed cryogenic spice grinding system yields better quality of black pepper powder under cryogenic conditions (at -50°C) as compared to that at ambient conditions (³10°C) in terms of average particle size, colour attributes, essential oil components, piperine, total phenolics and DPPH scavenging capacity.

Key words : Black pepper, cryogenic grinding, chemical properties, colour.

Introduction

Black pepper (*Piper nigrum* L.) is referred as the king of spices and belongs to the Piperaceae. It is one of the most important spices of India and world due to its use for food and medicinal purpose and its pungent flavour and chemical characteristics. Its fruits, known as berries, are dark green in colour which becomes bright orange and red when ripe. After sun drying, its colour changes from grayish to dark brown. It is consumed in the form of whole, cracked, coarse or medium or fine powder and oleoresin (Barnwal *et al.,* 2017; Meghwal and Goswami, 2013).

Grinding is one of the most common unit operations used to produce black pepper powder for consumption. Most chemical components and the effective substances of black pepper are heat sensitive substances (Liu *et al.,*2018). In ambient grinding of spices, temperature rises to the extent of 42 - 93°C as grinding is the most power consuming operation because only 1% of the energy imparted into the material is utilized for loosening the bond between particles, whereas almost 99% of input energy is dissipated as heat, rising the temperature of the ground product etc. The rise in temperature causes about 30-40% loss of volatile oil and flavouring constituents; for high oil bearing material, oil comes out from oil bearing material during grinding process, which makes ground product gummy, sticky and results in chocking of processing sieves through which the product passes (Singh and Goswami, 1999; 2000). The loss of volatile oil can be significantly reduced by cooling the black pepper seeds before feeding to the grinder and by maintaining low temperature in the grinding zone. A cryogenic grinding technique may be used to significantly reduce the loss of volatile oil, moisture, and colour. Liquid nitrogen at -195.6°C provides the refrigeration needed to pre-cool the spices and maintain the desired low temperature by absorbing

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the heat generated during the grinding operation. Cryogenic grinding of spices is used in order to obtain high quality products (Liu *et al.,* 2018, Ghodki and Goswami, 2017; Barnwal *et al.,* 2014). Cryogenic grinding minimally damaged chemical quality of the spices e.g. the lipid, crude protein, starch, non-volatile ether extract, piperine, essential oil and the typical pepper essential oil compounds (Liu *et al.,* 2018).

In this paper, an experimental study was conducted on cryogenic spice grinding system, at varying conveyor screw speed and varying temperature, for black pepper grinding and quality attributes of black pepper powder were investigated.

Materials and methods

Sample preparation

The black pepper seeds were procured from local market of Ludhiana, India. Cleaning of the seeds was done manually for removal of all foreign matter, dust, dirt, broken and immature seeds. The initial moisture content of the seeds was determined by vacuum oven method (temperature 70 °C and pressure 100 mm Hg) until a constant mass was obtained (Singh and Goswami, 2000) and it was 8.3% dry basis (db).

The experiments were conducted at ICAR-Central Institute of Post-Harvest Engineering and Technology (CIPHET), Ludhiana, India. The cryogenic spice grinding system, employed for the grinding of black pepper seeds, consists of a pre-cooling unit, grinder unit, spice powder collection unit, grading unit (sieving system) and a control panel to monitor and control the grinding operation (Barnwal *et al.,* 2018; Saxena *et al.,* 2018). The pre-cooling unit comprises of screw conveyor assembly, self-pressurized liquid nitrogen (LN_2) cylinder, and power transmission systems (Barnwal *et al.,* 2018). Using different temperatures (10°C and -50°C) and different conveyor screw speeds (5, 10 and 15 rpm), the grinding operation of black pepper was carried out in the cryogenic spice grinding system at 6500 rpm of pin mill (grinder unit). After grinding, all the black pepper powder samples were packed in sealed, moisture free and water proof flexible polythene bags for further analysis of quality parameters.

Particle size of black pepper powder

The particle size of black pepper powder was determined in duplicates using a vibratory sieve shaker with a set of Bureau of Indian Standards (BIS) sieves. Average particle size (D_p) of black pepper powder was computed using following expression (Sahay and Singh, 2004):

Average particle size, $D_P = \left[\, 0.135 \times \left(1.366\right)^{FM} \right]$ (1)

where F.M. is fineness modulus.

Colour attributes of ground black pepper powder

Colour values (*L*, *a* and b) of black pepper powder were determined by using Hunter Colorimeter (model no. 45/0 L, U.S.A). '*L*' is referred as lightness and extends from 0 (black) to 100 (white) whereas other two coordinates '*a*' and 'b' indicates redness (+*a* values) to greenness (-*a* values) and yellowness (+*b* values) to blueness (-*b* values), respectively. Hue angle (h^0) is characteristic of the colour by means of which the colour is perceived. Chroma value (C^*) is the characteristic of colour used to represent the degree of departure of the colour from gray of same lightness. Browning index (BI) represents the intensity of pure brown colour. Hue angle (h^0), chroma value (C^*) and browning index (BI) were calculated by using the following equations (Gupta *et al.,* 2011):

$$C^* = \sqrt{\left(a^2 + b^2\right)} \qquad (2)$$

$$h^0 = \tan^{-1}\left(\frac{b}{a}\right) \qquad (3)$$

$$BI = \left[\frac{100(x - 0.31)}{0.17}\right] \qquad (4)$$

where $$x = \left[\frac{(a + 1.75)}{(5.645L + a - 3.012b)}\right] \qquad (5)$$

Chemical properties

Essential oil

Essential oil (EO) was extracted from black pepper ground at variable screw speed (5, 10 and 15 rpm) and variable temperature (10°C and -50°C). Sixty grams seeds from each treatment were ground in a domestic mixer-grinder (Sujata, India). Ground samples were subjected to hydro distillation for 6 h using Clevenger apparatus (Clevenger, 1928). After decanting and drying of the oil over anhydrous sodium sulphate the corresponding mild yellow coloured oil were recovered and calculated in terms of percentage (V/W) and the EO yield was expressed in percentage. The colour of EO remained unchanged after two hours of extraction. This EO was analysed to identify the chemical constituents.

Gas chromatography–mass spectrometry (GC-MS) analysis of Essential oil

Analyses of essential oil samples were performed on an Agilent Technologies 7820A Series gas chromatograph coupled to Agilent 5975 C mass selective detector. One micro litres of EO was mixed with hexane in the ratio 1:1000 and injected to a HP 5 MS column (Agilent, USA, 30m, 0.250 mm film thickness 0.25µm) with the help of an auto sampler (Agilent 7693). Helium was used as the carrier gas at 1.0 ml min^{-1} flow rate with split ratio of 10:1. The oven temperature was programmed from 50°C for 3 min followed by incremental rate at 10°C min^{-1} to 180°C and 45°C min^{-1} to 280°C. The injector and the GC-MS interface temperatures were maintained at 250°C. Mass spectra were recorded at 70eV with mass range from m/z 50 to 550 amu. Authentic standards of major constituents of black pepper EO were run alone and in combination to get retention time of each constituent. Retention indices of all the constituents were determined by Chemstation software (Agilent technologies, USA). All samples were analyzed in triplicate.

Identification of compounds

The chromatograms were analysed for constituent C_5-C_{24} compounds on the basis of their RI (Retention Index), RT (retention time) and GC-MS library obtained on a non polar HP-5 MS column by comparison of RI, provided in the literature (Adams, 2007; McLafferty, 2009) and by comparison of mass spectra with those mentioned in NIST-MS (National Institute of Standards and Technology), mass spectral library of the GC-MS data system and co-injection with authentic compounds.

Extraction of fatty oil

Thirty gram seed powder was utilized for oil extraction with hexane using Accelerated Solvent Extraction System (M/s Dionex India Pvt. Ltd.). This technique accelerates the traditional extraction process by using solvent at elevated temperatures and pressures. Pressure is maintained in the sample cell to maintain the heated solvent in a liquid state during the extraction process. After heating, the extract is rinsed from the sample cell into a collection vessel. The solvent containing oil was concentrated under vacuum in a rotary evaporator (M/s JSGW, Ambala Cantt, India) at 35°C. Solvent free extract were weighed to determine the quantity and then transferred to amber coloured glass vials and stored in refrigerator until further analysis.

Total phenolic content

All the ground black pepper samples (10 g) were extracted with 50 ml methanol twice. Supernatant from both extraction were pooled and methanol was evaporated in rotary evaporator. This crude extract was used for determination of the total phenol and antioxidant activities. Total phenol concentrations were determined using a Folin-Ciocalteu assay, as described by Amin *et al.* (2006). An aliquot of 0.1 ml from 1,000 ppm crude methanol extract was taken in a test tube and made the volume 1 ml by adding solvent. Three ml of 10 % sodium carbonate was added. Previously 10-fold diluted Folin-Ciocalteu reagent was added to the mixture. The mixture was allowed to stand at room temperature for 90 min and then absorbance was measured at 710 nm. Gallic acid was used as the standard phenol. The amount of phenolic content was calculated by using the standard curve of Gallic acid having R^2 value ranged from 0.96 to 0.99 and was expressed as mg Gallic Acid Equivalents g^{-1} crude seed extract.

Piperine from the powdered sample was extracted by refluxing in alcohol and estimated by Shimadzu High Performance Liquid Chromatography (HPLC) equipped with SPD-10A UV-visible detector. The column used was Reverse Phase C-18 with a size of 4.6 × 250 mm. Acetonitrile and 1% acetic acid (48:52) were the mobile phase with a flow rate of 1.5 ml min^{-1} and measurement was taken at 342 nm. Percentage piperine was computed by using authentic standard (Wood *et al.,* 1988).

DPPH scavenging capacity

The antioxidant activity of crude seed extracts was evaluated on the basis of its activity in scavenging the stable DPPH radical using the method described by Shimada *et al.* (1992). Crude seed extract (10^5ppm) was diluted in respective solvent to give at least 5 different concentrations. An aliquot (1, 1.5, 2, 2.5 ml) of the extract of each concentration was mixed with 1ml of 1M DPPH solution. The mixture was then homogenized and left to stand for 30 min in the dark. The absorbance was measured at 517 nm against a blank of methanol using a spectrophotometer. DPPH solution plus respective solvent was used as control and Butyl Hydroxyl Toluene (BHT) (0-80ppm) was used as a standard reference synthetic antioxidant with R^2 value ranging from 0.95- 0.99. Results were expressed as mg BHT Equivalent g^{-1} crude seed extract. The per cent scavenging effect was calculated from equation (6).

Scavenging effect (%) =

$$\frac{A_{517}\text{ of control} - A_{517}\text{ of extract}}{A_{517}\text{ of control}} \times 100 \qquad (6)$$

Statistical analysis

Analysis of variance for quality characteristics were carried out using LSD of AgRes Statistical software (Version 3.01, Pascal International Software Solution, USA).

Results and discussion

Average particle size of black pepper powder

The variation of average particle size of black pepper powder with conveyor screw speed and temperature is represented in Table 1. The average particle size increased with increasing conveyor screw speed irrespective of temperature. The reason may be that due to increase in screw speed; there will be increase in feed rate and hence lower retention time of seeds in grinder. The increased feed rate may be resulted in pushing out the coarser particles (increased average particle size) from the shearing area (grinding unit) due to higher input of black pepper seed to shearing area. In addition, a higher particle size was obtained at 10°C irrespective of conveyor screw speed in comparison to that of at -50°C. This is so because in ambient grinding, heat generation resulted in temperature rise and therefore the moisture and oil content in the spice sample remains in liquid state. It leads to a sticky mass if further grinding continues and therefore restriction of smaller particle size (Singh and Goswami, 1999; Meghwal and Goswami, 2010) and production of larger particle size of black pepper. However, from Table 1, it is clear that average particle size of black pepper powder varied non-significantly with studied conveyor screw speed under both conditions (at 10°C and -50°C).

Colour parameters

Table 2 represents the variation of colour parameters i.e. L, a, b, hue angle, chroma value and browning index with screw speed and temperature. From Table 2, it is clear that L –value varied non-significantly with screw speed under both conditions (at 10°C and -50°C). The variation of *a, b,* chroma value, hue angle and browning index were significant ($p \leq 0.05$) with varying speed at -50°C and that of non-significant with varying speed at 10°C. It was reported that a more bright and light colour (more L and less BI values) of black pepper powder was obtained at -50°C in comparison to that at 10°C (Table 2). Meghwal and Goswami (2010) reported that a light and vivid powder obtained due to preservation of brightness and natural lust of powder under cryogenic grinding conditions. The cryo-ground black pepper samples were observed better in colour (Barnwal *et al.,* 2017). When grinding at 10°C,

Table 1. Effect of screw speed and temperature on particle size of black pepper powder

S.No.	Average particle size (mm)		
	Screw speed (rpm)	Temperature	
		10°C	-50°C
1	5	0.345±0.004	0.313±0.007
2	10	0.359±0.018	0.327±0.034
3	15	0.369±0.005	0.343±0.010
	F-values	4.31^{NS}	0.73^{NS}
	$CD_{0.05}$	0.04	0.06

NS=Non-significant

Table 2. Effect of screw speed and temperature on colour parameters of black pepper powder

Temp.	Screw speed rpm	*L*-value	*a*-value	*b*-value	Chroma value	Hue angle (°)	Browning index
10°C	5	47.67±2.46	2.97±0.41	9.78±1.11	10.23±1.06	72.92±3.02	27.31±3.89
	10	50.61±2.27	3.02±0.58	8.81±2.49	9.40±2.17	69.17±6.29	23.40±5.95
	15	50.88±3.45	2.50±0.60	10.33±1.59	10.65±1.59	76.26±3.28	26.34±5.92
	F-values	3.29^{NS}	2.27^{NS}	1.42^{NS}	1.14^{NS}	2.39^{NS}	1.15^{NS}
	$CD_{0.05}$	3.89	0.56	1.90	1.74	7.73	5.56
-50°C	5	52.14±1.39	$3.23±0.30^{a}$	$10.11±0.61^{ab}$	$10.61±0.62^{a}$	$72.24±1.52^{a}$	$25.77±2.32^{a}$
	10	53.69±2.67	$2.19±0.44^{b}$	$9.41±0.94^{b}$	$9.67±0.96^{b}$	$76.92±2.31^{b}$	$22.01±3.16^{b}$
	15	50.79±2.94	$2.34±0.56^{b}$	$10.49±0.80^{a}$	$10.76±0.81^{a}$	$77.41±2.95^{b}$	$26.29±3.73^{a}$
	F-values	2.82^{NS}	12.72^{*}	3.74^{*}	4.25^{*}	11.89^{*}	4.44^{*}
	$CD_{0.05}$	3.83	0.46	0.83	0.85	2.43	3.25

NS=Non-significant; Mean values with the same superscript letters within the same column do not differ significantly (p > 0.05) the powder lost its brightness and turns into dark colour due to more rise in powder temperature.

Essential oil components

Figures 1a and b show the variation of some major essential oil components in black pepper powder samples, identified through GC-MS analysis. The variations of average thujene (%), camphene (%), myrcene (%) and α–phellandrene (%) with temperature and conveyor screw speed are depicted in Fig.1a. Thujene (%), camphene (%), myrcene (%) and α–phellandrene (%) of black pepper samples were lower than that of seed. The maximum values of thujene (1.27 %), camphene (0.07 %), myrcene (1.17 %) and α–phellandrene (0.57 %) of black pepper samples were obtained at -50°C (10 rpm and 15 rpm), -50°C(15 rpm), -50°C(10 rpm), and -50°C(10 rpm), respectively (Fig.1a). At 10°C, minimum values of thujene (1.13 %), camphene (0.05 %), myrcene (0.94 %) and α–phellandrene (0.4 %) of black pepper samples were obtained at 10 rpm, 15 rpm, 10 rpm and 15 rpm, respectively. Liu *et al.* (2018) reported that for black pepper ground by ambient grinding (hammer mill), camphene (0.18 %) and α–phellandrene (6.22 %) whereas for black pepper ground by cryogenic grinding, camphene (0.22 %) and α–phellandrene (0.19 %) were obtained. Figure 1b represents the variation of average α–pinene (%), sabinene (%), β–pinene (%) and D-limonene (%) at different temperature and conveyor screw speed conditions. It was found that α–pinene (%), sabinene (%), β–pinene (%) and D-limonene (%) of seed were higher than that of black pepper samples. The maximum values of α–pinene (4.37 %), sabinene (12.16 %), β–pinene (6.89 %) and D-limonene (18.85 %) of black pepper samples were obtained at -50°C (15 rpm), -50°C(15 rpm), -50°C (15 rpm), and -50°C (10 rpm), respectively (Fig.1b). At 10°C, minimum values of α–pinene (3.30 %), sabinene (10.98 %), β–pinene (5.35 %) and D-limonene (15.62 %) of black pepper samples were obtained at 10 rpm, 15 rpm, 10 rpm and 15 rpm, respectively. Liu *et al.* (2018) observed that for black pepper ground by cryogenic grinding, α–pinene (5.20 %), sabinene (1.84 %) and β–pinene (6.57 %) whereas for black pepper ground by ambient grinding (hammer mill), α–pinene (5.20 %), sabinene (0.47 %) and β–pinene (8.90 %) were obtained.

Total phenolic content and DPPH scavenging capacity

Figures 2a and b show the variation of average piperine (%), total phenol (%) and DPPH scavenging activity in black pepper powder samples. The variations of percent piperine and percent total phenol (alcohol extract and water extract) with temperature and conveyor screw speed are represented in Fig.2a. The maximum values of piperine (2.65 %), total phenol-alcohol extract (1.00 %) and total phenol-water extract (0.87 %) of black pepper samples were obtained at -50°C and 5 rpm (Fig.2a). At 10°C, minimum values of piperine (2.34 %), total phenol-alcohol extract (0.95 %) and total phenol-water extract (0.71 %) of black pepper samples were obtained 5 rpm, 5 rpm and 15 rpm, respectively. The values of piperine and percent total phenol (alcohol extract and water extract) were higher for black pepper samples, obtained at -50°C than that at 10°C for same conveyor screw speed (Fig.2a). The antioxidant activity of crude seed extracts was evaluated on the basis of its activity in scavenging the stable DPPH radical using the standard method (Shimada *et al.,* 1992). Figure 2b shows the variations of percent DPPH activity (alcohol extract and water extract) with temperature and conveyor screw speed. The maximum values of DPPH

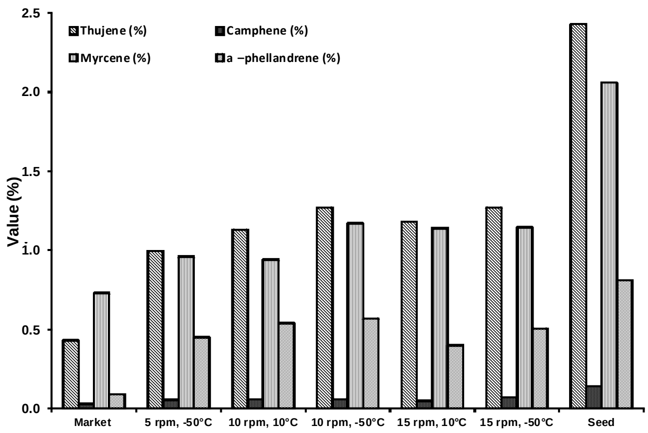

Fig. 1a. Variation of Thujene, Camphene, Myrcene and ~-phellandrene of black pepper powder with screw speed and temperature

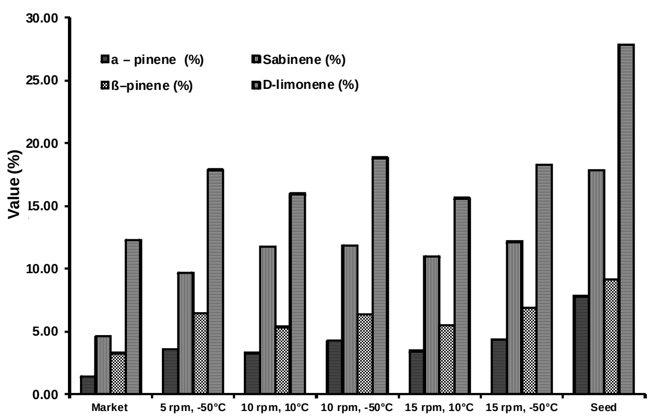

Fig. 1b. Variation of ~- pinene, Sabinene, -pinene and D-limonene of black pepper powder with screw speed and temperature

activity-alcohol extract (23.85 %) and DPPH activity-water extract (22.66 %) of black pepper samples were obtained at -50°C and 5 rpm (Fig.2b). At 10°C and 15 rpm, minimum values of DPPH activity-alcohol extract (19.40 %) and DPPH activity -water extract (18.89 %) of black pepper samples were obtained. The values of DPPH activity-alcohol extract and DPPH activity-water extract of black pepper samples were higher for black pepper samples, obtained at -50°C than that at 10°C for same conveyor screw speed (Fig.2b). The black pepper powder, obtained at -50°C grinding condition, retained higher amount of the piperine, percent total phenol (alcohol extract and water extract) and DPPH activity (alcohol extract and water extract) than that obtained at 10°C grinding condition (Figs 2a and b). Phenolics are quite heat unstable and reactive compounds (Cheynier, 2005). During grinding at 10°C, there is temperature rise leading to reduction in phenols whereas grinding at -50°C improves the phenolic content. In cryogenic grinding (at -50°C), the vaporization of liquid nitrogen to the gaseous state, creates an inert and dry atmosphere which finally reduces the loss of quality attributes of spices (Singh and Goswami, 1999). Saxena *et al.* (2012) also observed higher phenolic content and antioxidant content in cryo-ground coriander and fenugreek genotypes. Cryogenic grinding ensured the highest quality of pepper products (Liu *et al.,* 2018).

Conclusions

From present study, it was concluded that the black pepper powder, obtained at cryogenic grinding conditions (-50°C), was better than that obtained at ambient conditions ($\geq$10°C), in terms of average particle size, colour attributes, essential oil components, piperine, total phenolics and DPPH scavenging capacity. Average particle size, obtained at 10°C, was more than that of at -50°C irrespective of conveyor screw speed. The variation of *a, b*, chroma value, hue angle and browning index were significant ($p \leq 0.05$) with varying speed at –50°C and that of non-significant with varying speed at 10°C. It was observed that a more bright and light colour (more L and less BI values) of black pepper powder was obtained at -50°C in comparison to that at 10°C. The maximum values of piperine (2.65 %), total phenol-alcohol extract (1.00 %) and total phenol-water extract (0.87 %) of black pepper samples were obtained at -50°C and 5 rpm. The maximum values of DPPH activity-alcohol extract (23.85 %) and DPPH activity-water extract (22.66 %) of black pepper samples were obtained at -50°C and 5 rpm.

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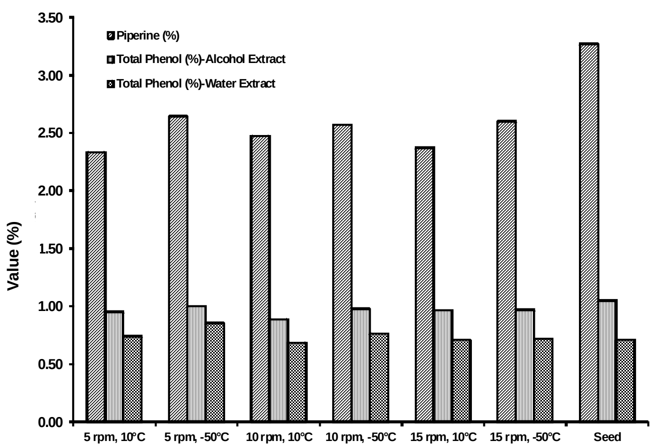

Fig. 2a. Variation of piperine and total phenolic content of black pepper powder with screw speed and temperature

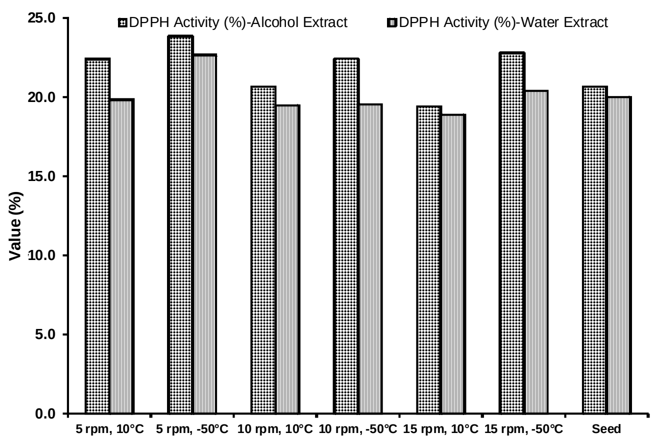

Fig. 2b. Variation of DPPH activity of black pepper powder with screw speed and temperature

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