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Performance evaluation of cryogenic spice grinding system for fenugreek powder production

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Abstract

This paper presents the performance evaluation of cryogenic spice grinding system for quality improvement of fenugreek powder. Fenugreek seed was employed to ground at two temperatures (10°C and -50°C) with varying conveyor screw speed (5, 10 and 15 rpm), at 6500 rpm pin mill speed. The powder, obtained at -50°C, contained higher amount of volatile oil (3.88 ± 0.13%), total phenolics (48.48 ± 3.43 mg GAE g^{-1}), flavonoids (75.01 ± 0.36 mg QE g^{-1}), and antioxidant (33.98 ± 1.04 mg BHTE g^{-1}) and these were found significantly ($p \leq 0.05$) higher than the corresponding values of fenugreek powder, obtained at 10°C grinding. The particle size was significantly affected ($p \leq 0.05$) due to variation in temperature and screw speed and its value varied from 0.277 ± 0.002 to 0.352 ± 0.009 mm. Brighter and lighter coloured ground fenugreek was obtained at -50°C grinding. Significantly higher liquid nitrogen was consumed (0.53 – 0.91 kg kg^{-1} of seeds) at -50°C grinding than that at 10°C grinding (0.31-0.39 kg kg^{-1} seeds). Net specific energy consumption was ranged from 37.30 to 63.22 kJ kg^{-1} of fenugreek seeds with the variation in both temperature and screw speed. It was observed that developed cryogenic spice grinding system yields better quality of fenugreek powder under cryogenic conditions (at -50°C) as compared to that at 10°C or higher temperature (ambient conditions).

Introduction

Fenugreek (*Trigonella foenum-graceum* L.), also known as 'methi' (Chaturvedi and Pant, 1988), is an annual herb of leguminosea. It is being used as spice with its seeds and as vegetable with its fresh tender pods, leaves and shoots. As a spice, it flavours food. It is one of the principal constituent of curry powder. In India, fenugreek is used as a lactation stimulant (Tiran, 2003). It contains high protein (25%), lysine (5.7g / 16g N), soluble (20%) and insoluble (28%) dietary fibre besides being rich in calcium, iron, and β-carotene (NIN, 1987). Fenugreek seeds are referred to have hypoglycemic and hypocholesterolemic effects in human beings (Khosla *et al.*, 1995). The seeds have a strong aroma and somewhat bitter taste. The seeds are very hard and difficult to grind (Kakani *et al.*, 2009).

Grinding is one of the most common unit operations and it is used to produce spice seed powder for various end uses. In grinding, only 1% of the energy imparted into the material is utilized for loosening the bond between particles, and almost 99% of input energy is dissipated as heat, resulting in temperature rise of the ground product. In conventional or ambient grinding of spices, there is temperature rise of 42-95°C. This temperature rise leads to the loss of volatile oil and flavouring constituents. For high oil bearing material, oil comes out from oil bearing material during grinding and it leads to ground product gummy, sticky and thus chocking of sieves occurs while the product passes through the sieves (Singh and Goswami, 1999; 2000). By using a cryogenic grinding technique, the loss of essential constituents, moisture, and colour can be significantly reduced. For pre-cooling the spice and maintaining the desired low temperature by absorbing the heat generated during the grinding operation, liquid nitrogen at -195.6°C provides the required refrigeration. Liu *et al.* (2018) has reported that 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.

Considering the disadvantages of conventional grinding, there is a need for cryogenic grinding of spice in order to

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obtain high quality ground spice products. In the present communication, attempts were made to evaluate the performance of cryogenic spice grinding system for fenugreek powder production.

Materials and methods

Sample preparation

The fenugreek seeds were taken from ICAR-NRCSS, Ajmer, India. The seeds were manually cleaned for removing all foreign matter, dust, dirt, broken and immature seeds. Its initial moisture content was ascertained by using vacuum oven method (70 °C and 100 mm Hg pressure i.e. 13.33 kPa) until a constant mass was attained (Kakani *et al.*, 2009; Singh and Goswami, 2000) which was 7.4% dry basis (db) .

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

Liquid nitrogen and specific energy consumption

The LN_2 consumption was determined as the ratio of weight difference of LN_2 dewar before grinding (Wi) and after grinding (Wf) to the weight of the feed (Ws) and expressed as:

$$LN_2 \text{ consumption (kg kg}^{-1} \text{ of seed)} = \frac{W_i - W_f}{W_s} \quad (1)$$

Power consumption (ΔW) during grinding operation was calculated by following expression:

$$\text{Power consumed, } \Delta W = W_{OL} - W_{NL} = V \times (I_{OL} - I_{NL}) \quad (2)$$

where, V is operational voltage of operation and I_{OL} and I_{NL} are the current recorded at on load and no load conditions, respectively.

Feed rate (f) was calculated as a ratio of weight of the feed to the time consumed during the operation of grinder.

Specific energy consumption (ΔE) was determined by using the following equation (Singh and Goswami, 1999):

$$\Delta E = \frac{\text{Power consumed (W)} \times 3.6}{f\,(kg/h)} = \frac{DW \times 3.6}{f} \quad (3)$$

Particle size of fenugreek powder

Particle size of the fenugreek powder was determined in duplicates using a vibratory sieve shaker with a set of Bureau of Indian Standards (BIS) sieves. Average particle size of fenugreek powder was computed by using the following equation (Sahay and Singh, 2004):

$$\text{Average particle size, } D_p = [0.135 \times (1{\cdot}366)^{FM}] \quad (4)$$

where FM is fineness modulus.

Colour attributes of ground fenugreek powder

Colour (L, a and b) values of the ground fenugreek were determined by using Hunter Colorimeter (model no. 45/0 L, U.S.A). The values of hue angle (h^0), chroma value (C^*) and brownness index (BI) were computed by using the following standard formulae (Gupta *et al.*, 2011):

$$C^* = \sqrt{(a^2 + b^2)} \quad (5)$$

$$h^0 = \tan^{-1}\left(\frac{b}{a}\right) \quad (6)$$

$$BI = \left[\frac{100(x-0.31)}{0.17}\right] \quad (7)$$

$$\text{where, } x = \left[\frac{(a+1.75)}{(5.645L+a-3.012b)}\right] \quad (8)$$

Chemical properties

Essential oil

From ground fenugreek samples, essential oil was extracted by hydro distillation using Clevenger apparatus lighter than water type and oil was quantified as volume by weight percentage (Saxena *et al.*, 2012; Barnwal *et al.*, 2018).

Total phenolic content

Total phenol concentrations were determined using a standard Folin-Ciocalteu assay (Amin *et al.*, 2006). All the ground fenugreek 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 utilized for determination of the total phenol and flavonoids concentration, as well as antioxidant activities. The standard procedure has been used for determination of phenolic content and Gallic acid was used as the standard phenol (Saxena *et al.*, 2015). Phenolic content was expressed as mg Gallic Acid Equivalents/g crude seed extract (mg GAE/g seed extract).

Total flavonoid content

Total flavonoid concentration was determined by using standard method (Saxena *et al.*, 2015) Quercetin has been used as the standard flavonoids. The amount of flavonoid was expressed as mg Quercetin Equivalents/g crude seed extract (mg QE/g crude seed extract), as per procedure followed by Saxena *et al.* (2015).

Antioxidant content

The antioxidant content of crude extract was determined on the basis of its activity in scavenging the stable DPPH radical using the standard method (Shimada *et al.*, 1992). The standard procedure (Saxena *et al.*, 2015) was employed for determination of percent scavenging effect. DPPH solution plus methanol was taken as control and that Butyl hydroxyl toluene (BHT) as a standard reference synthetic antioxidant. The absorbance, measured at 517 nm using a spectrophotometer. Antioxidant content was expressed as mg Butyl hydroxyl toluene Equivalent/g crude seed extract (mg BHTE / g crude seed extract).

The percent scavenging effect was computed as follows (Saxena *et al.*, 2015): Scavenging effect (%) =

$$\frac{A_{517}\text{ of control} - A_{517}\text{ of extract}}{A_{517}\text{ of control}} \times 100 \qquad (9)$$

Statistical analysis

Analyses of variance (ANOVA) for the various characteristics were carried out using LSD of AgRes Statistical software (Version 3.01, Pascal International Software Solution, USA).

Results and discussion

Liquid nitrogen and specific energy consumption

A higher amount of liquid nitrogen was utilized at -50°C grinding (0.53–0.91 kg kg^{-1} of seeds) as compared to the consumption at 10°C grinding (0.31-0.39 kg kg^{-1} seeds) irrespective of screw speed. The specific energy consumptions varied from 37.30 to 63.22 kJ kg^{-1} fenugreek seeds with variation of temperature and screw speed. Singh and Goswami (1999) reported that specific energy consumption increased from 55 to 98 kJ kg^{-1} with increasing grinding temperature from -160°C to -70°C for cryogenic grinding of cumin seed. The specific energy consumption (kJ kg^{-1}) was observed to decrease with increase in screw speed irrespective of temperature. This may be due to lower retention time of seeds in grinder and hence lower specific energy consumption (kJ kg^{-1}) with increased feed rate at higher screw speeds (Meghwal and Goswami, 2010). At low grinding temperature, the degree of brittleness of spice seed increased and hence required less energy in grinding (Singh and Goswami, 1999).

Average particle size of fenugreek powder

The average particle size of fenugreek powder varied significantly with screw speed and temperature (Table 1). The average particle size increased with increasing screw speed irrespective of temperature. Increasing screw speed results in increased feed rate and lower retention time of seeds in grinder. The increased feed rate may be resulted in pushing out the coarser particles as more fenugreek seed entered the shearing area and coarser grinding (increased average particle size) at higher screw speeds took place. In addition, a higher particle size was obtained at 10°C irrespective of screw speed. This is so because at ambient grinding, heat generation resulted in temperature increase and the moisture and oil content in the spice sample remains in liquid state which leads to a sticky mass if further grinding continues. This results in

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

S.No.	Average particle size (mm)		
	Screw speed (rpm)	Temperature	
		10°C	-50°C
1	5	0.327±0.004a	0.277±0.002a
2	10	0.349±0.007b	0.294±0.010a
3	15	0.352±0.009b	0.314±0.011b
	F-values	11.03*	13.19*
	CD0.05	0.01	0.02

**p≤0.05; NS-non significant; n=2; Mean values with the same superscript letters within the same column do not differ significantly (p > 0.05).*

restriction of smaller particle size (Singh and Goswami, 1999; Meghwal and Goswami, 2010).

Colour parameters

From Table 2, it is clear that the L –value (indicator of lightness) varied significantly with screw speed under both conditions (at 10°C and -50°C). The variation of a, b, chroma value and hue angle were non-significant with varying speed at 10°C and that of significant with varying speed at -50°C. It was observed that a brighter and lighter coloured fenugreek powder was obtained at -50°C. In cryogenic grinding (-50°C), a light and vivid powder obtained due to preservation of brightness and natural lust of powder (Meghwal and Goswami, 2010). However, for grinding at 10°C, there is more rise in temperature powder which turns into dark in colour and lost its brightness.

Essential oil

One of the quality parameters is essential oil content. It is a measure of aroma and flavour in the spice powder. For spice powder having higher volatile oil content, its market value will be higher in financial terms as spices are valued for their aroma and flavour (Gopalakrishnan *et al.*, 1991). The essential oil content of fenugreek powder was found to be varied non-significantly with screw speed (Table 3) whereas significant with the grinding temperature. In cryogenically ground fenugreek powder

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

Temp.	Screw speed (rpm)	L-value	a-value	b-value	Chroma Value	Hue angle (°)	Browning index
10°C	5	68.67±2.31a	4.52±0.47	35.11±0.80	35.40±0.81	82.66±0.73	72.48±3.49
	10	71.55±1.52b	4.36±0.40	33.57±2.76	34.58±1.90	82.71±1.02	67.79±5.30
	15	71.23±1.46b	4.29±0.69	34.39±1.14	34.67±1.14	82.89±1.15	68.27±4.53
	F-values	4.69*	0.39NS	1.47NS	0.86NS	0.11NS	2.64NS
	CD0.05	1.41	0.56	1.86	1.42	1.42	4.69
-50°C	5	69.60±1.05a	5.57±0.51a	34.22±0.78a	34.68±0.82a	80.75±0.74a	72.82±5.42
	10	71.82±1.31b	4.37±0.27b	36.45±0.72b	36.71±0.73b	83.16±0.36b	72.04±2.84
	15	72.30±0.64b	4.28±0.26b	36.19±1.24b	36.44±1.23b	83.24±0.45b	72.44±4.87
	F-values	12.39*	30.21*	13.22*	10.60*	54.00*	0.07NS
	CD0.05	1.64	0.38	0.98	0.99	0.56	4.69

**p≤0.05; NS-non significant; n=2; Mean values with the same superscript letters within the same column do not differ significantly (p > 0.05).*

Table 3. Effect of screw speed and temperature on essential oil, total flavonoids, total phenolic content and antioxidant content of fenugreek powder

Temp.	Screw speed (rpm)	Essential oil (%)	Total flavonoids (mg QE/g crude seed extract)	Total phenolic content (mg GAE/g seed extract).	Antioxidant content (mg BHTE / g crude seed extract).
10°C	5	2.70±0.80	54.55±0.14	21.23±2.16a	22.38±3.52
	10	2.72±0.36	61.23±1.88	24.87±4.51b	24.27±0.59
	15	2.69±0.28	67.29±6.16	25.61±1.56b	24.55±0.32
	F-values	0.07NS	0.98NS	40.87*	0.59NS
	CD0.05	2.61	14.41	3.06	9.28
-50°C	5	3.61±0.14	56.04±0.79a	31.51±1.44a	27.39±2.78a
	10	3.88±0.13	68.29±4.4b	34.44±2.52a	33.19±0.62b
	15	3.76±0.52	75.01±0.36b	48.48±3.43b	33.98±1.04b
	F-values	0.66NS	24.48*	26.98*	22.86*
	CD0.05	1.02	11.15	11.26	4.57

**p≤0.05; NS-non significant; n=2; Mean values with the same superscript letters within the same column do not differ significantly (p > 0.05).*

(-50°C), higher values of essential oil content was observed as compared to fenugreek, ground at 10°C (Table 3). At high temperatures, the volatile oils evaporate from spices. The reason may be that at 10°C, mass transfer increased due to increase in vapour pressure which resulted in a loss of volatile oil. The results are in agreement with the findings about a significant increase in volatile oil content of cryo-ground coriander of different genotypes (Saxena *et al.*, 2012; 2015).

Total flavonoids, phenolic content and antioxidant content

The total flavonoids, total phenolic content and antioxidant content were found to increase slightly with increase in screw speed (Table 3). It may be due to high feed rate which resulted in lower residence time in grinder leading to retention of these medicinal constituents. Gopalakrishnan *et al.*, (1991) reported similar observations for ambient grinding of cardamom. As compared to fenugreek powder (obtained by grinding at 10°C temperature), the fenugreek powder (obtained by grinding at -50°C temperature) retained higher amount of the total flavonoids, total phenols and antioxidant content, respectively (Table 3). Cheynier (2005) mentioned that phenolics are quite heat unstable and reactive compounds. During grinding at 10°C, there is temperature rise resulting 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 eventually reduces the loss of quality parameters of spices (Singh and Goswami, 1999). Saxena *et al.* (2012) also reported the higher flavonoid content, phenolic content and antioxidant content in cryo-ground coriander and fenugreek genotypes. Liu *et al.* (2018) has observed and reported that cryogenic grinding ensured the highest quality of pepper products.

Conclusion

The fenugreek powder, obtained at cryogenic grinding conditions (-50°C), was better than that at 10°C, in terms of quality parameters such as essential oil, total phenolics, flavonoids, antioxidant and particle size. The fenugreek powder, obtained with grinding at -50°C, had volatile oil (3.88±0.13%), total phenolics (48.48±3.43 mg GAE g^{-1}), flavonoids (75.01±0.36 mg QE g^{-1}), and antioxidant (33.98±1.04 mg BHTE g^{-1}) and these were found significantly ($p \leq 0.05$) higher than the corresponding values of fenugreek powder, obtained at 10°C grinding. The particle size was significantly affected ($p \leq 0.05$) due to variation in temperature and screw speed. Its value varied from 0.277±0.002 to 0.352±0.009 mm. The net specific energy consumption ranged from 37.30 to 63.22 kJ kg^{-1} of fenugreek seeds.

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