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Optimizing volatile oil retention in ajwain (*Trachyspermum ammi* L.) through cryogenic grinding techniques

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Abstract

The therapeutic benefits of seed spices have long been acknowledged, and there is growing interest in their potential biological activity in human health. Ajwain (*Trachyspermum ammi* L.), a significant seed spice from the Apiaceae family, is characterized by its distinct aroma and taste, attributed to its volatile oil content (2.5-5%). In postharvest processing, grinding is a crucial stage requiring careful handling to preserve flavor and aroma constituents. However, conventional grinding methods can lead to volatile oil loss due to varying grinding temperatures. To address this, a study was conducted using cryogenic grinding of ajwain seeds with liquid nitrogen on a laboratory-scale cryogenic grinder. The grinding was performed at temperatures ranging from 0°C to -100°C, with feed rates of 8, 9, and 10 kg/h, and sieves of 0.8 and 1.0 mm. The study focused on liquid nitrogen consumption and the volatile oil content of the ground powder. The optimal conditions for producing high-quality ajwain seed powder were determined to be a feed temperature of -60°C, a feed rate of 8 kg/h, and a sieve size of 0.8 mm, achieving the highest retention of volatile oil at 2.9%. This approach highlights the effectiveness of cryogenic grinding in preserving the volatile oil content in ajwain seeds, offering a promising method for maintaining the quality of spice powders.

Key words: Spices, ajwain, cryogenic grinding, volatile oil.

Introduction

Spices are vital in global agriculture due to their high economic value and diverse applications. Ajwain (*Trachyspermum ammi* L.), a member of the Apiaceae family, was originally cultivated in Egypt and India but is now grown in dry regions such as Iran, Pakistan, and Afghanistan. Ajwain seeds are extensively used in India and other parts of Asia for culinary and medicinal purposes. This significant crop is prized for its volatile oil, utilized in the food industry for its flavoring properties. Ajwain oil boasts various medicinal attributes, including antibacterial, antifungal, anti-inflammatory, and antioxidant effects (Rao, and Sowbhagya, 2017). The seeds are renowned for their digestive and antiseptic qualities and are commonly employed in traditional medicine to alleviate digestive issues such as indigestion, flatulence, and diarrhea. Additionally, ajwain seeds possess stimulant, carminative, and antispasmodic properties. Key components of ajwain oil, thymol and carvacrol, exhibit potent antibacterial and antifungal properties, making the oil a valuable food preservative against microbial spoilage (Hanif *et al*., 2021; Asangi *et al*., 2023).

Grinding spices postharvest is crucial for preserving their aroma and flavor. Ground spices can be utilized directly or processed into value-added products such as mixes, oleoresins, and spice oil extracts, which find extensive use in the food, cosmetics, perfumes, personal hygiene, and pharmaceutical industries (Das *et al*., 2018; Wani *et al*., 2022). The primary objective of grinding spices is to achieve a smaller particle size while maintaining excellent flavor and color. However, the grinding process can elevate the product's temperature to 42-95°C, depending on the mill and spice, leading to a significant loss of aroma and flavor. Minimizing this loss is vital to maximize the profit of spice processors (Aradwad *et al*., 2021).

Temperature plays a crucial role in retaining the oil content during the grinding of ajwain seeds (Sardar *et al.*, 2018). Cryogenic grinding, involving liquid nitrogen, has been shown to preserve more volatile oil and maintain a higher quality in the resulting powder. This method offers substantial economic benefits to entrepreneurs and consumers by enhancing the market value of ajwain. According to Badrkhani *et al*. (2013), ajwain seeds contain approximately 2-4% volatile oil, with thymol comprising 40-65%. Thymol is widely used in cosmetics for its fragrance, food additives for flavoring, and traditional medicines as an active ingredient (Aradwad *et al*., 2021). Additionally, it is a key component in producing perfume chemicals and is frequently included in mouthwashes due to its efficacy in killing oral bacteria.

The objective of this study was to explore advanced grinding techniques, such as cryogenic grinding, to enhance the quality and profitability of ajwain powder, thereby making it a viable and attractive option for the spice industry.

Materials and methods

The entire study was conducted at the College of Food Processing Technology and Bio Energy, Anand Agricultural University in Anand, India.

Samples of uniform ajwain seeds were obtained from the Nathwani Group of Companies in Jamnagar, Gujarat, India. To limit the loss of volatile oil, ajwain seeds were packed in aluminum laminated pouches and stored at room temperature.

Experimental Design: A complete randomized design (factorial) was adopted for the for the experiment (Steel and Torie, 1980). ANOVA and correlations were performed with Design Expert 10 statistical software.

Cryogenic grinding of the ajwain seed: The cryogenic grinding system used in this study comprised several key components. The system included a large tank with a capacity of 185 cubic meters to hold liquid nitrogen, maintained at a pressure of 2 MPa. A stainless steel feed hopper with an adjustable shutter controlled the rate at which the material was fed into the system. Directly below the hopper, a rotary valve facilitated the movement of the material towards the cryogenic screw conveyor.

The cryogenic screw conveyor, measuring 80 cm in length, featured a special auger made of stainless steel. This auger transported the material and mixed it with liquid nitrogen to achieve the desired grinding temperature. Liquid nitrogen flowed continuously until the material reached its glass transition temperature.

Grinding was performed using a pin mill operating at a fixed speed of 4,500 rpm. Post-grinding, the material was collected in a separate hopper. The control panel of the system included three on/off switches for the rotary valve, pin mill, and screw conveyor, as well as a knob for adjusting the speed of the cryogenic screw conveyor. An emergency stop knob was also available. The system was equipped with a digital temperature controller for setting the grinding temperature and a digital indicator displaying voltage, current, and frequency information.

The cryogenic grinding of ajwain seeds was conducted in three replicates for each treatment. The parameters included three feed rates (8, 9, and 10 kg/h), six grinding temperatures (0, -20, -40, -60, -80, and -100 °C), and two sieve sizes (0.8 and 1.0 mm).

Determination of volatile oil content: The Clevenger apparatus was used to obtain volatile oil from ajwain seeds via steam distillation. First, 50 g of ajwain seed powder was weighed and added to a flask. Approximately 500 mL of distilled water and several antifoaming beads were also added to the flask. The mixture was then brought to a boil, and a reflux rate of one to two drips per second was maintained. Refluxing continued until the oil volume in the traps remained constant over two consecutive readings taken at 1-hour intervals. The collected samples were then transferred to vials and allowed to cool to room temperature.

The percentage of volatile oil (on a moisture-free basis, mL/100 g) was calculated using the following equation:

$$\text{Volatile oil (\%)} = \frac{\text{Volume of volatile oil (mL)}}{\text{Dry weight of sample (g)}} \times 100$$

Measurement of liquid nitrogen (LN2) consumption: The consumption of liquid nitrogen (LN2) for grinding varies depending on the temperature and other parameters. To accurately measure LN2 consumption, the LN2 cylinder was placed on a weighing scale with a capacity of 500 kg. The weight of the cylinder was recorded both before and after grinding the sample. The difference in these weight readings represented the consumption of LN2 in kilograms.

Results and discussion

Effect of temperature, feed rate, and sieve size on volatile oil content: The volatile oil content served as a reliable indicator of the quality of spice powder, which typically diminished significantly during conventional grinding. In this study, the volatile oil content of cryogenically ground ajwain seed powder increased from 2.0% to 3.6% as the grinding temperature was reduced from 30°C to -100°C. This improvement was attributed to the reduced heat generation in the grinding mill at lower temperatures, minimizing the loss of volatile oil.

The cryogenic grinding parameters influenced the powder's volatile oil content: temperature, feed rate, and sieve size. Table 1 illustrated that temperature (0 to -100°C) significantly affected the volatile oil content, with the calculated F value exceeding the tabulated value, indicating a significant effect at the 5% level. The interactions between temperature, feed rate, and sieve size also impacted the volatile oil content, though to a lesser extent than the individual parameters, suggesting that these interactions were not as significant.

Effect of temperature and feed rate on volatile oil content: The relationship between feed rate and temperature on the volatile oil content is depicted in Fig. 1. When the temperature of the ajwain seeds decreased from 0 to -100°C, the volatile oil content in the powder increased from 2.5% to 3.6%. This result was likely due to reduced heat generation during grinding at lower temperatures, thereby minimizing volatile oil degradation. These findings were consistent with previous studies on black pepper by Meghwal and Goswami (2010) and Singh and Goswami (1999), which concluded that cryogenic grinding helped reduce the loss of volatile oil from spices.

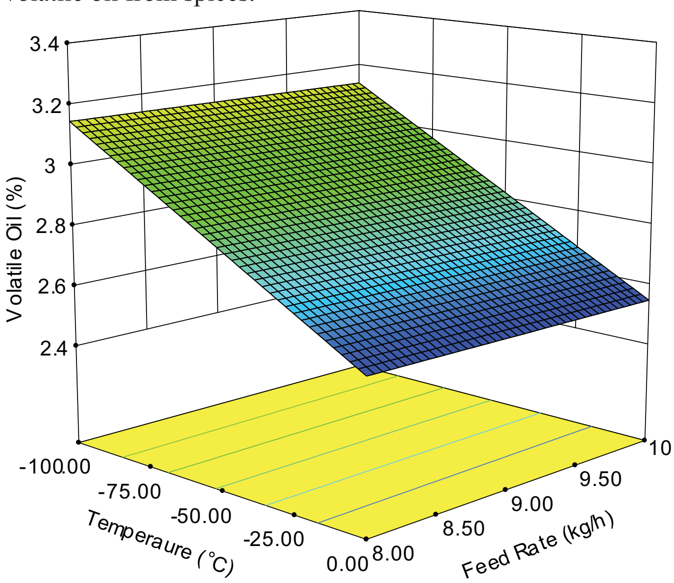

Fig.1. Effect of temperature and feed rate on volatile oil content

Additionally, as the feed rate increased from 8 to 10 kg/h, the volatile oil content in the powder also increased. This may have been attributed to the reduced grinding time associated with a higher feed rate. A shorter grinding duration meant the material spent less time in contact with the pin mill, resulting in less heat generation and better retention of volatile oil. Murthy *et al*. (2014) supported this hypothesis, stating that a slower feed rate increased the grinding temperature, reducing the volatile oil percentage in the powder, whereas a faster feed rate enhanced the volatile oil content.

Effect of temperature and sieve size on volatile oil content: The effect of temperature and sieve size on the volatile oil content of ajwain seed powder is illustrated in Fig. 2. Both sieve size

Table 1. ANOVA for effect on volatile oil content

Source	MS	F Cal	F Tab	Test	CD (5%)	CV (%)
T	43.172	132.26	2.10	*	0.378	10.49
F	0.214	0.66	3.95	NS	NS	
T*F	3.200	9.80	1.87	*	0 .656	
S	0.570	1.75	3.96	NS	NS	
T*S	1.870	5.73	2.10	*	0.535	
F*S	0.539	1.65	3.95	NS	NS	
T*F*S	1.711	5.24	1.87	*	0.928	
Error	0.326					

and temperature significantly affected the volatile oil content. As shown, when the sieve size increased from 0.8 to 1.0 mm, the volatile oil content in the powder also increased. This could be due to less cell rupturing within the seed microstructure, as the larger mesh size facilitated easier powder flow. Similarly, a decrease in grinding temperature from 0°C to -100°C resulted in increased volatile oil content in the powder. Similar results on the effects of temperature during the cryogenic grinding of cloves were reported by Singh and Goswami (2000), where they found a reduction in volatile oil content at higher temperatures when the sieve size was 0.5 mm.

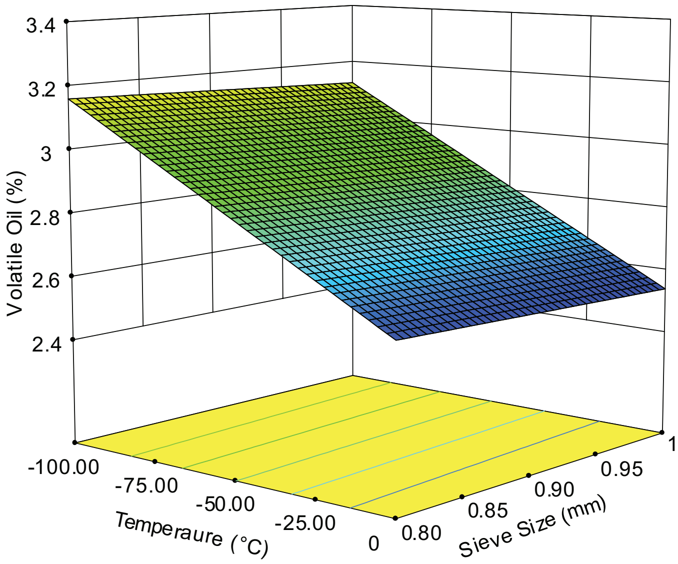

Fig. 2. Effect of temperature and sieve size on volatile oil content

Overall, the study indicated that optimizing the cryogenic grinding parameters-specifically temperature, feed rate, and sieve size-significantly enhanced the retention of volatile oil in ajwain seed powder, improving its quality and market value.

Effect of feed rate and sieve size on volatile oil content: Fig. 3 illustrates the effect of sieve size and feed rate on the volatile oil content in the powder. Both parameters significantly influenced the volatile oil content. The volatile oil content also increased as the feed rate increased from 8 to 10 kg/h and the sieve size increased from 0.8 to 1.0 mm. These results indicate that using finer sieve sizes maximizes volatile oil retention.

Effect of temperature, feed rate and sieve size on LN_2 consumption: Procuring and storing liquid nitrogen (LN_2) required significant care and attention. The system could not be completely sealed because LN_2 begins to boil at -196°C and continuously evaporates from the vessel. Therefore, LN_2 needed to be used immediately after procurement to minimize losses.

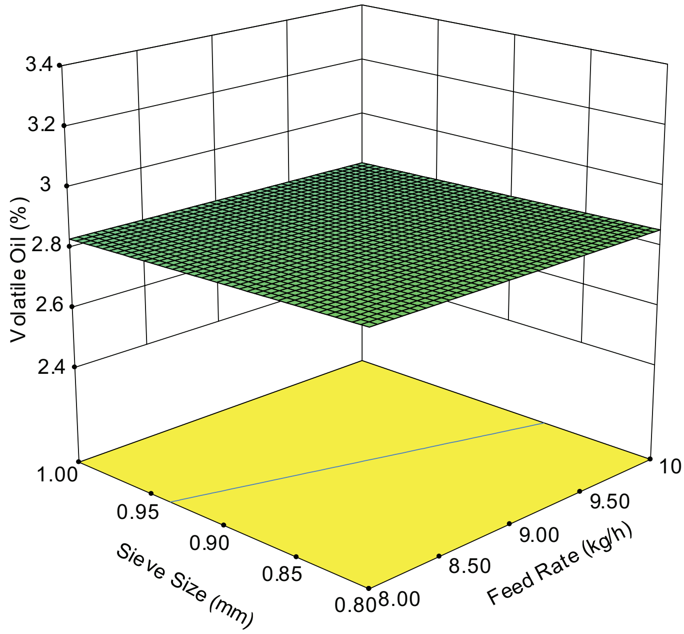

Fig.3. Effect of feed rate and sieve size on volatile oil content

During the cryogenic grinding of ajwain seeds, as the temperature of the seeds decreased from 0 to -100°C, the amount of LN_2 consumed increased from 2.4 to 7.1 kg. Similar studies conducted by Singh and Goswami (2000) for cryogenic grinding of cloves.

The ANOVA results in Table 2 demonstrated a significant effect of temperature (-0 to -100°C) on LN_2 consumption. The calculated F value exceeded the tabulated value, indicating a significant effect at the 5% level. There were also significant interactive effects between temperature, feed rate, and sieve size on LN_2 consumption. However, the interactions involving feed rate and sieve size alone were insignificant, as their lower F values indicated.

Table 2. ANOVA for effect on LN2 consumption

Source	MS	F Cal	F Tab	Test	CD (5%)	CV(%)
T	18.462	136.06	2.35	*	0.245	9.771
F	0.002	0.02	3.13	NS	NS	
T*F	4.369	32.20	1.97	*	0.425	
S	0.001	0.01	3.98	NS	NS	
T*S	2.170	15.99	2.35	*	0.347	
F*S	1.080	7.96	3.13	*	0.141	
T*F*S	0.649	4.78	1.97	*	0.601	
Error	0.135					

Effect of temperature and feed rate on LN_2 consumption: Fig. 4 illustrates the relationship between temperature and feed rate on LN_2 consumption. As the temperature decreased from 0 to -100°C, LN_2 consumption significantly increased for all feed rates. This increase is due to the greater liquid nitrogen required to achieve lower temperatures for the ajwain seeds. These findings are consistent with those reported by Meghwal and Goswami (2010) for the cryogenic grinding of black pepper.

Conversely, increasing the feed rate from 8 to 10 kg/h decreased LN2 consumption across all grinding temperatures. This decrease may be attributed to the reduced grinding time at higher feed rates, which means the material requires less time to reduce its size, thus consuming less liquid nitrogen. Ghodki and Goswami (2016) conducted similar studies on the effect of

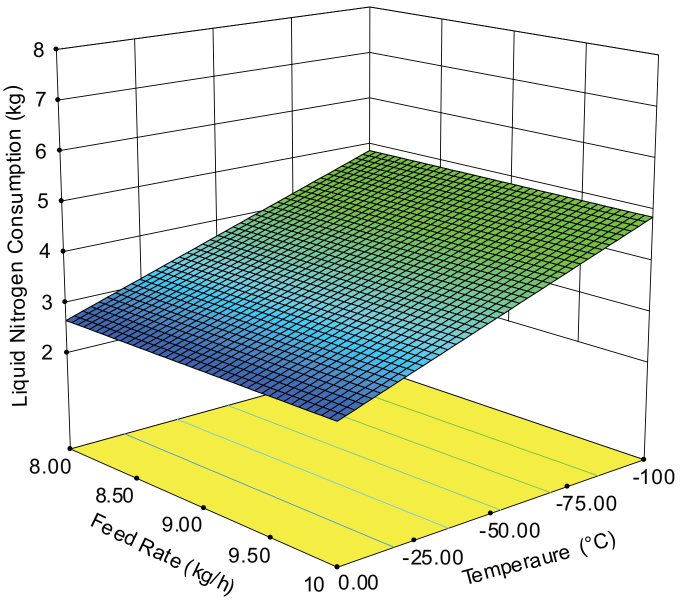

Fig. 4. Effect of temperature and feed rate on LN_2 consumption

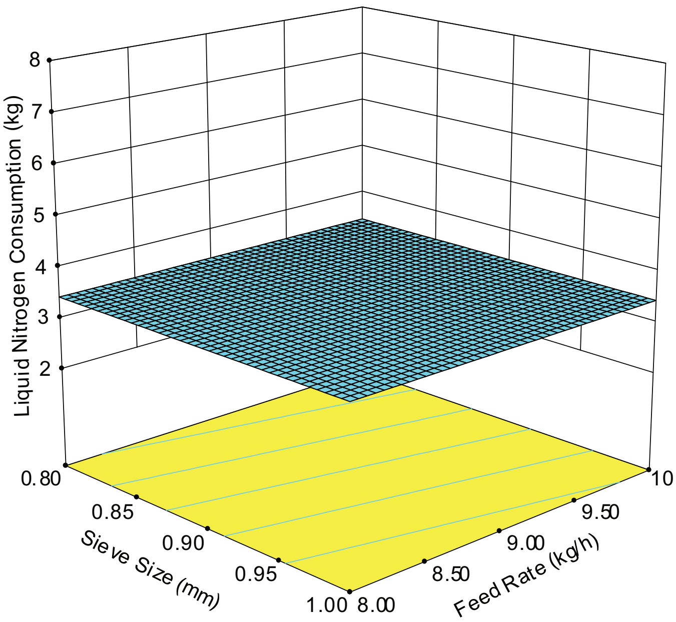

Fig.6. Effect of feed rate and sieve size on LN_2 consumption

grinding temperatures on black pepper powder's particle and physicochemical characteristics.

Effect of temperature and sieve size on LN_2 consumption: The effect of temperature and sieve size on LN_2 consumption is shown in Fig. 5. Both sieve size and temperature had a substantial impact on LN_2 consumption. As the sieve size increased from 0.8 to 1.0 mm, LN_2 consumption decreased. This is likely because coarser material production requires less time in pin mills compared to finer material. Additionally, as the temperature decreased from 0 to -100°C, LN_2 consumption increased gradually from 2.4 to 7.2 kg.

Effect of feed rate and sieve size on LN_2 consumption: The effect of feed rate and sieve size on LN_2 consumption during grinding is depicted in Fig. 6. Both parameters significantly affected LN_2 consumption. As the feed rate increased from 8 to 10 kg/h and the sieve size increased from 0.8 to 1.0 mm, the requirement for liquid nitrogen decreased. This reduction is likely due to the shorter grinding time required for materials at higher feed rates and larger sieve sizes.

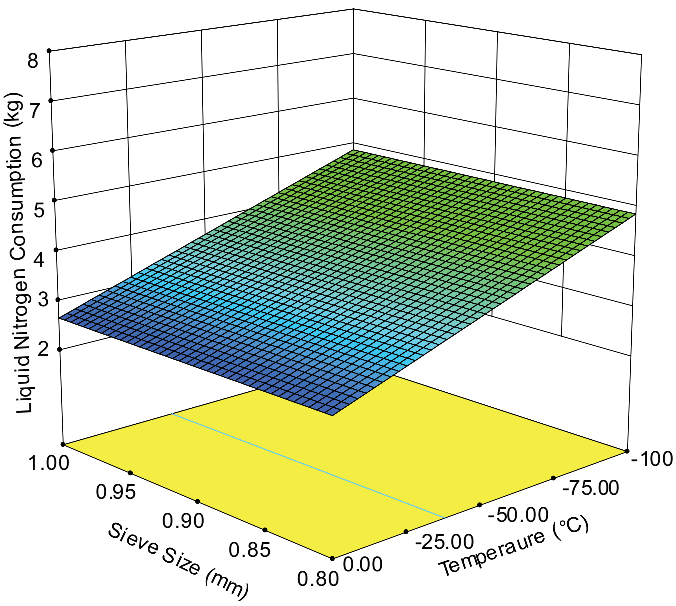

Fig.5. Effect of temperature and sieve size on LN_2 consumption

Optimization of processing parameters for cryogenic grinding of ajwain seed powder: The dependent parameters influencing the cryogenic grinding of ajwain seed powder included grinding time, energy consumption, liquid nitrogen (LN_2) consumption, and volatile oil content. To produce superior quality ajwain powder, optimizing these parameters to achieve minimum grinding time, minimum energy consumption, minimum LN2 consumption, and maximum oil content are volatile. The optimized conditions determined for this study were a cryogenic grinding temperature of -60°C, a feed rate of 8 kg/h, a sieve size of 0.8 mm, and a volatile oil content of 2.9%.

This study demonstrated the effectiveness of cryogenic grinding for retaining volatile oil in ajwain seeds. The results showed that feed rate and sieve size had a significant impact on volatile oil content, with finer sizes resulting in maximum oil retention. Liquid nitrogen consumption increased with lower grinding temperatures, highlighting the need for careful management of LN2 usage. Overall, cryogenic grinding proved to be a valuable technique for preserving the quality of volatile oils in ajwain seeds.

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