



# An Overview of Cryomilling within the Realm of Spices

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## Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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## ABSTRACT

Cryogenic milling, a method that utilizes extremely low temperatures to grind materials, shows great potential for the processing of spices within the food industry. This article offers an examination of cryogenic milling in the context of spice processing, emphasizing its possible advantages and obstacles. Through the preservation of the aroma and volatile compounds found in spices, cryogenic milling provides benefits such as improved retention of flavor, reduced particle size, and improved product quality. Nevertheless, it also has drawbacks such as high equipment costs, high energy consumption, safety issues, and difficulties related to moisture levels and particle size distribution. Despite these challenges, cryogenic milling holds promise for producing top-quality spices with enhanced flavor profiles. Future research and development should concentrate on refining cryogenic milling techniques, addressing safety and regulatory concerns, and exploring potential applications in emerging food products and markets. In general, cryogenic milling is a

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valuable technology for spice processing and has the capacity to contribute to the creation of innovative and high-quality food products.

*Keywords: Cryomilling; spices; nitrogen; gas; oil.*

## 1. INTRODUCTION

Cryomilling, also known as cryogenic grinding or cryogrinding, is an innovative technique revolutionizing the field of food processing. Cryogenic gases, viz. liquid nitrogen or argon, are used to cool food materials to extremely low temperatures before milling [1]. This process creates a brittle state in food particles, facilitating their size reduction into fine powders or particles with enhanced properties. Cryomilling has become increasingly popular in the food processing industry because it may solve problems with conventional milling techniques such as heat generation, volatile component loss, and ingredient degradation. Cryomilling is very useful for processing delicate or heat-sensitive components since it maintains the sensory qualities, nutritional content, and functional qualities of food products while processing at cryogenic temperatures [2].

Various foodstuffs like coffee, cocoa, chocolates, coconut, dehydrated meat products, tea etc. can be Cryomilled. Freeze-grinding is carried out by injecting controlled amounts of liquid nitrogen into the grinding zone of the spices [3]. The rapid evaporation of the liquid nitrogen quickly cools both the spice and mill. It simultaneously absorbs frictional heat generated during from grinding. The temperature of the grinding zone is reduced to below -100°F while in conventional milling the temperature of the spice to during grinding reaches to 200°F. Cryomilling allows extremely fine grinding due to solidification of spices oils to brittleness at these low temperatures. These ultrafine spice powders disperse the flavor evenly throughout the end product and eliminates specking defects. The low temperature of the freeze-grinding process also reduces the loss of aroma and moisture. The ground products retain most of their original flavor, strength and weight. On average, 7 g of the freeze-ground spices have the same flavor power as 10 g of their conventional milled counterparts. Evaporation of liquid nitrogen in the grinding zone and removes any air from the mill, which reduces the oxidation of spice oils. Freeze-ground spices are much more stable than traditional ground spices. Grinding rates increase

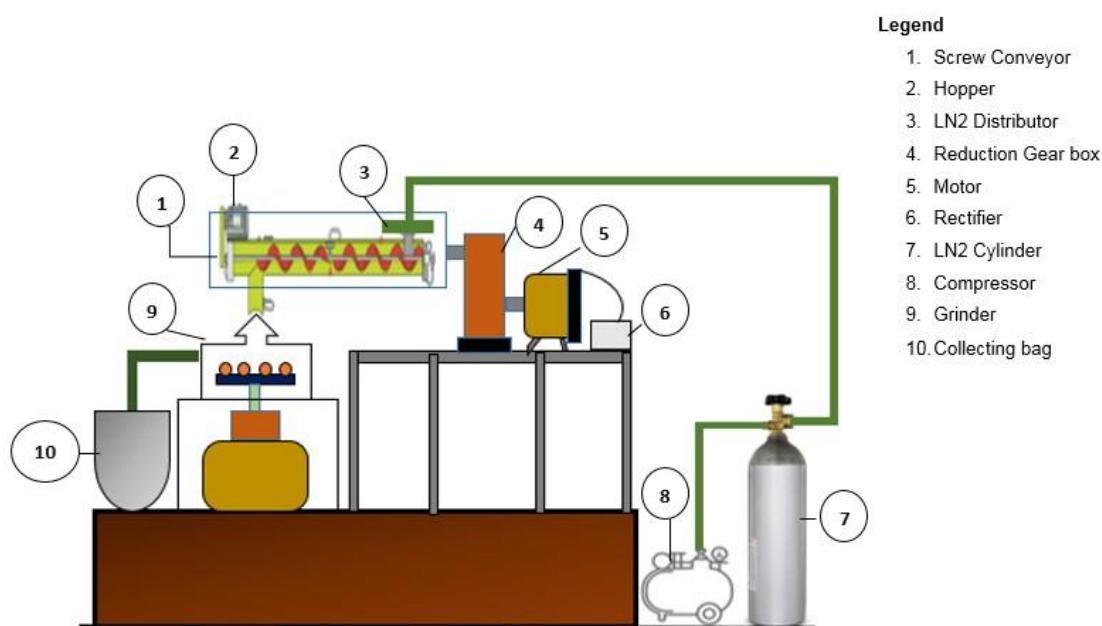
as the mill's low-temperature operation reduces the amount of 'gumming up' on grinding surfaces and screens [4].

## 2. CRYOMILLING STUDIES

In a study, the impacts of sieve size (0.8 mm, 1.0 mm, and 1.5 mm), feed rate (5 kg/h, 6 kg/h, and 7 kg/h) and grinding temperature (-30°C, -20°C, -10°C, 0°C, and 10°C), on grinding time, mean particle size, and volatile oil content of cumin seeds was evaluated. The optimized parameters for cryogenic grinding of cumin powder were: sieve size of 0.8 mm, feed rate of 7 kg/h and temperature of -30°C. The volatile oil content in the ground sample was 3.27% having 29.61% cuminaldehyde and 5.36%  $\gamma$ -terpinene [5].

This study focused on the impact of cryogenic grinding on various properties of seed extracts from two ajwain genotypes. The results showed that there was an increase in essential oil and oleoresin recovery in cryoground seeds of both genotypes. The effect of cryogrinding was less pronounced in genotype AA 93 than in genotype AA 2. Dimethyl sulfoxide (DMSO) and hexane were found to be the most effective solvents for extracting flavonoid and phenolic contents from ground seeds. The cryoground samples exhibited a greater DPPH scavenging percentage and antioxidant content, indicating strong antioxidant activity. The crude seed extract (methanol) derived from the cryo-ground samples from genotype AA-2 possessed highest total phenolic content, while that of genotype AA 93 had the maximum TPC. The crude seed extract (hexane) of genotype AA 2 had the highest total flavonoid content. The total antioxidant content of the seed extracts of the cryo-ground samples also increased, with no significant variation observed between the genotypes. Both the AA 2 and AA 93 genotypes exhibited the highest antioxidant content. [6].

Sharma et al. [7] focused on the impact of cryogenic grinding on various components of seed extracts from two cumin genotypes, namely, oleoresin, volatile oil, total phenolics, antioxidant properties and flavonoid content. The results revealed that cryogenic grinding effectively preserved volatiles in both genotypes

**Legend**

1. Screw Conveyor
2. Hopper
3. LN2 Distributor
4. Reduction Gear box
5. Motor
6. Rectifier
7. LN2 Cylinder
8. Compressor
9. Grinder
10. Collecting bag

**Fig. 1. Cryomilling system**

and even enhanced the recovery rate. In contrast, noncryogenic or normal grinding at room temperature led to a significant loss of volatile oil, amounting to 18-19% in both genotypes. Interestingly, the use of cryogenic grinding resulted in a remarkable increase of 28.28% in oleoresin content for the RZ 209 genotype. However, the increase was comparatively lower at 16.046% for the GC4 genotype.

Barnwal et al. [8] examined cryogenic and ambient grinding conditions to determine the grinding characteristics of turmeric and cinnamon. A pin mill laboratory grinder was used for the spice grinding process. Sieve analysis was conducted to determine various grinding characteristics of the ground spices. Compared with those of cryogenic ground spices, mixtures of ambient ground spices exhibited greater average volume, particle size, surface mean diameter, volume mean diameter, mass mean diameter, and specific surface area. Under cryogenic conditions, the average particle sizes of ground turmeric and cinnamon were 0.336 mm and 0.356 mm, respectively. Under ambient conditions, the average particle sizes of ground turmeric and cinnamon were 0.407 mm and 0.454 mm, respectively. The energy constants and specific energy consumption during cryogenic grinding were lesser than those during ambient grinding. For turmeric, the Rittinger's and Kick's constants for cryogenic ground

powder were 34.8 and 39.7 KWh/tonne, respectively, and while for ambient ground powder they were 58.0 and 58.1, respectively. The color of cryogenic ground spices was superior to that of ambient ground spices. Overall, the grinding characteristics of turmeric and cinnamon were greater under cryogenic than under ambient grinding conditions.

Murthy, et al., [9] The current obstacles in conventional grinding techniques have been identified. Conventional grinding equipment such as hammer mills and roller grinders has proven to be efficient in manufacturing top-notch powders across different sectors. Nevertheless, these machines tend to reduce moisture levels because of the high temperatures produced during grinding. Conversely, cryogenic grinding utilizing liquid nitrogen functions at extremely low temperatures. This groundbreaking method can be utilized in a flexible way by integrating different coolants as refrigerants.

Murthy et al. [10] studied pilot-scale grinding of black pepper and reported that a control sample subjected to ambient grinding reached product temperatures of 86 and 53°C at feed rates of 15 and 35 kg hr<sup>-1</sup>, respectively. This indicated that a low feed rate would result in higher temperatures, which is considered undesirable. To address this issue, the product temperature was controlled and maintained within a range of -15 to -60°C using liquid nitrogen, while the feed

rates ranged from 7 to 60 kg hr<sup>-1</sup> as per the experimental design. The average volatile oil content after cryogenic grinding was found to be 1.7 mL/100 g, whereas after ambient grinding, it was only 0.9 mL/100 g. Notably, the loss of volatile oil after ambient grinding was approximately 50% greater than that after cryogenic grinding.

The impact of cryogenic grinding of nine different coriander (*Coriandrum sativum* L.) on the volatile oil content, oleoresin content, total phenolic content, flavonoid content, and antioxidant properties of seed extracts genotypes was examined. The oleoresin content in the cryogenically ground samples was significantly greater, volatile oil ranging from 0.14% (genotype RCr 436) to 0.39% (genotype Sindhu) and oleoresin from 13.80% (genotype ACr 1) to 19.58% (genotype Sindhu). The yield of crude seed extract (methanol) was consistently greater in cryogenically ground samples across all genotypes, with the total phenolic content also being elevated in all genotypes, ranging from 32.44 (RCr 41) to 92.99 mg GAE/g (Sindhu genotype). Similarly, the total flavonoid content was greater in all cryogenically ground samples, ranging from 15.28 (Sindhu genotype) to 20.85 mg quercetin equivalent (QE)/g (Swati genotype). DPPH scavenging activity was consistently greater in cryogenically ground seeds across all genotypes, indicating better antioxidant activity in cryogenically ground samples. In conclusion, cryogenic grinding technology can preserve the flavor and antioxidant activities of coriander regardless of the genotype [11].

Saxena et al. [11] standardized the grinding procedure and reported that the application of energy to break down particles into smaller particles resulted in the generation of heat. This increase in temperature within the grinder reached a maximum of 95°C. As a consequence, the increase in temperature caused a loss of approximately 30% of the volatile oil and led to the production of a dark-colored powder. The extremely low temperature of the grinder caused the oils to solidify, resulting in the spices becoming more brittle and easily crumbled. This, in turn, allowed for a finer and more consistent grinding process. The dried fruits of coriander seeds exhibited varying essential oil contents, ranging from 0.1% to 0.33%.

Meghwal and Goswami [12] studied the impact of ambient and cryogenic grinding on black pepper.

The findings from a comparative analysis showed that ambient grinding has higher power requirement (8.92%) and specific energy (14.5%) than cryogenic grinding. Moreover, particle size analysis revealed that cryogenic grinding yielded coarser particles. Furthermore, the investigation of the constant energy law illustrated that ambient grinding consumes more power. In terms of quality, cryogenic grinding led to a higher volatile oil content (2.15 ml/100 g) and a powder with superior freshness but higher yellowness (14%) and lower whiteness (40%) indices.

Bhadravathi [13] studied on cryogenic grinding of turmeric and observed the increase in specific energy consumption from 55 to 94 kJ/kg as the grinding temperature increased from -60 to -8°C. For grinding temperatures ranging between 55 and 60°C, the specific energy consumption ranged from 174 to 181 kJ/kg, following a polynomial relationship of second-order. Additionally, at lower grinding temperatures, there was an increase in brittleness, leading to reduced energy consumption during grinding.

Beera, [14] The examination of the process of grinding cumin powder in a controlled environment was conducted in great detail, and enhancements were implemented to improve the sensory characteristics and longevity of the ground cumin. Initially, the product temperatures of conventionally ground cumin seeds reached a maximum of 95°C. Unfortunately, this elevated temperature caused significant losses of volatile oil and alterations in volatile constituents. However, by incorporating a technique that involved circulating chilled water (10-25°C) in the grinding area, the temperature was effectively reduced. As a result, there was better preservation of volatile oil, a finer particle size, and improved sensory properties of the ground cumin. In terms of optimal packaging and storage, it was determined that aluminum-LDPE laminated pouches were highly suitable. These pouches maintained the quality of the cold ground cumin when stored at 37°C and 70% relative humidity, providing a shelf life of 119 days.

Peter [15] described mature cumin seeds that were used for producing oil, either in their entirety or in a coarsely ground state. The process of extracting essential oil involves hydrodistillation, also known as steam distillation, which results in a potent aroma and a colorless or pale-yellow oily liquid. The yield of oil varied

between 2.5% and 4.5%, depending on whether the distillation was performed using whole seeds or coarsely ground seeds. To preserve the volatile oil, it was stored in securely sealed bottles or aluminum containers.

Singh and Goswami [16] conducted a study on clove grinding and identified the effective grinding temperatures for clove to below  $-50^{\circ}\text{C}$ , with no residues left on the sieve surface. Increasing the temperature within the cryogenic range ( $-110$  to  $-50^{\circ}\text{C}$ ) did not have a significant impact on the volatile oil content. Conversely, elevating the temperature within the ambient range ( $55$  to  $85^{\circ}\text{C}$ ) led to a notable decrease in the volatile oil content from  $11.0$  to  $9.3$  mL/100 g. In comparison, powder samples ground cryogenically contained 29.5% more volatile oil than those ground under ambient conditions.

Mckee et al. [17] performed grinding trials on cardamoms under four distinct temperature conditions: room temperature ( $28^{\circ}\text{C}$ ), overnight deep freezing the seeds ( $-18^{\circ}\text{C}$ ), dry ice grinding of seeds, and seeds precooling in liquid nitrogen. When the cardamom was ground at room temperature using a centrifugal mill with sieve openings of 1.00, 0.75, 0.50, and 0.25 mm, the losses of volatile oil and natural aroma ranged from 26% to 52%. However, alternative grinding techniques, such as the use of a deep freezer ( $-18^{\circ}\text{C}$ ), dry ice, and liquid nitrogen, significantly enhanced product quality based on the level of cooling. A premium quality product with high volatile flavor was obtained from cryogenic grinding with dry ice and liquid nitrogen in a centrifugal mill.

Singh and Goswami [18] conducted a study on the grinding of cumin seeds at various cryogenic and ambient temperatures. Their findings indicated that the volatile oil content was not significantly affected by a temperature increase within the cryogenic range ( $-160$  to  $-70^{\circ}\text{C}$ ). Conversely, an increase in temperature within the ambient range ( $40$  to  $85^{\circ}\text{C}$ ) resulted in a

notable decrease in volatile oil content from 2.86 to 2.26 mL/100 g. Furthermore, they observed that in the cryogenic grinding temperature range, the remained unchanged. However, increasing the temperature under ambient grinding conditions causes significant losses in the volatile oil components.

Spices with a high fat content can cause problems such as temperature rise and clogged sieves during the grinding process. Due to the increase in temperature, spices may lose a significant amount of their volatile oils and flavor components. To address this issue, a cryogenic grinding system was created to cool the spices before grinding and maintain a cryogenic temperature in the grinding zone. Experiments conducted on ground cumin seeds demonstrated that successful grinding could be accomplished at temperatures below  $-70^{\circ}\text{C}$ . However, once the temperature exceeds this limit, sieve clogging becomes an issue. Moreover, a noticeable rise in the specific energy consumption during grinding and the particle size of the final product was found when the grinding temperature was increased from  $-160^{\circ}\text{C}$  to  $-70^{\circ}\text{C}$ . However, the insignificant ( $p>0.05$ ) reduction in the volatile oil content from 3.30 to 3.26 ml/100 g was noted at the above temperature conditions [18].

Gopalkrishnan et al. [19] studied the grinding of cardamoms under four different conditions: ambient ( $28^{\circ}\text{C}$ ), overnight deep freezing ( $-18^{\circ}\text{C}$ ), dry ice grinding, and liquid nitrogen precooling of seeds. When the cardamom was ground at ambient temperature using a centrifugal mill with sieve openings of 1.00, 0.75, 0.50, and 0.25 mm, the losses of volatile oil and natural aroma ranged from 26% to 52%. However, depending on the extent of cooling, the grinding methods such as deep freezing ( $-18^{\circ}\text{C}$ ), dry ice, and liquid nitrogen, showed noticeable improvements in the product quality. The superior quality product with more highly volatile flavor constituents was obtained with cryogenic grinding with dry ice and liquid nitrogen in a centrifugal mill.

**Table 1. Essential oils of different ground spices**

Spice	Essential oil	
	Ambient	Cryogenic
<i>Black pepper</i>	1.40	1.84
<i>White pepper</i>	0.40	0.80
<i>Ginger</i>	1.44	1.80
<i>Coriander</i>	0.26	0.40
<i>Caraway</i>	2.96	4.40
<i>Celery</i>	1.70	2.25

Source: Wistreich & Schafer [22]

Pesek & Wilson [20] and Pesek et al. [21] studied the cryogenic and ambient grinding of Oregano, Nutmeg, Cinnamon, Cumin and White pepper. They displayed the results showing that the cryoground spices were better than the ambient ground spices due to: (a) cryogenic grinding can retain more of the light and volatile components of spices than ambient grinding; (b) while the both grinding techniques can very well retain the high-molecular-weight components.

Feature specific comparison of cryogenic and ambient grinding techniques (Annon 1962) [22,21,20,23]

- a) Cryogenic grinding effectively safeguards the aromatic properties and moisture content of spices while grinding, ensuring that the resulting ground products maintain their original flavor strength.
- b) By employing cryogenic grinding, the oxidation of spice oils is minimized, allowing for the preservation of flavor. The expulsion of air from the grinding zone due to evaporation of liquid nitrogen, facilitating oxidation free grinding. Moreover, low temperatures render spices brittle and solidify the oils, resulting in finely ground spices that evenly disperse flavor in the final products.
- c) Cryoground spices enhance the even distribution of flavor in liquid preparations, guaranteeing a consistently uniform taste throughout.
- d) Compared with conventionally ground products, cryoground spices exhibit superior stability. It is plausible that spices absorb or retain some nitrogen, which contributes to their enhanced stability.
- e) The operation of the mill at low temperatures during cryogenic grinding prevents the grinding surfaces and screens from becoming adhesive, thereby increasing the grinding rate.
- f) Cryoground spices offer a cost-effective solution, as they deliver superior flavor strength in comparison to conventional products. Furthermore, a finely ground texture and increased stability are advantageous for food processors.
- g) The cryogenic grinding process can be applied to a diverse range of foods beyond spices, including cocoa, coffee, tea, coconut, and dehydrated meat.

### 3. CONCLUSION

Cryogenic grinding of spices provides a promising technology with various advantages,

such as better flavor retention, finer particle size reduction, and enhanced product quality by subjecting spices to extremely low temperatures using cryogenic gases. Cryogenic grinding helps to maintain the delicate aroma and volatile compounds that contribute to the flavor profile of spices. Additionally, it allows for the production of spices with a finer particle size distribution, which can result in improved dispersion and flavor release in culinary applications, while it is crucial to recognize the constraints linked to cryomilling, including the substantial expense of equipment, energy usage, safety concerns, and difficulties pertaining to moisture levels and particle size distribution. These limitations emphasize the necessity for meticulous evaluation of aspects such as scalability, feasibility, and adherence to regulations when incorporating cryomilling into spice processing procedures. Cryomilling offers notable advantages in the field of spice processing. However, for effective use, a well-rounded approach that considers the advantages and disadvantages of this technology is needed. By means of ongoing research, innovation, and optimization efforts, cryomilling is a need of a day for the production of superior spices for a variety of culinary and food applications.

### 4. FUTURE PROSPECTS

Continued research and development efforts may lead to the optimization of cryomilling processes, resulting in increased efficiency and production. This could include improvements in milling machine design, process automation, and optimization techniques to increase throughput while retaining product quality. Loh et al., [24]. With advancements in biotechnology, personalized cryomilling solutions that are designed to suit the unique properties of various spices have potential. This may involve adjusting milling factors such as temperature, pressure, and duration to attain the best particle size distribution and flavor preservation for each type of spice. Kumar et al., [25].

The creation of innovative food products that require finely ground spices with an intact flavor and aroma may benefit from the use of cryomilling. Nutraceuticals, functional foods, and creative culinary creations that call for premium ingredients and processing methods may fall under this category. Technological developments in food safety and quality assurance could result in better cryomilling process monitoring and control systems. To guarantee constant product quality and safety, real-time monitoring of crucial

factors, including temperature, moisture content, and particle size distribution, may be needed. Expanding the application of cryomilling to emerging countries may present opportunities given the rise in globalization and the desire for a variety of gastronomic experiences. This could entail working together with regional food producers and spice producers to modify cryomilling technology to satisfy the particular needs and preferences of various cultures and geographical areas. Tiwary et al., [26].

## 5. LIMITATIONS

1. **Equipment Cost:** Cryomilling apparatuses, including cooling systems and cryogenic mills, can be costly to purchase and operate. The initial outlay and continuous running expenses can be too high for small-scale spice producers or companies with tight budgets.
2. **Energy consumption:** Liquid nitrogen and other cryogenic gases, which require considerable energy to be created and stored, are necessary for cryomilling. Compared to conventional milling techniques, cryogenic cooling may require more energy, particularly if optimization is not achieved.
3. **Safety Concerns:** Safety dangers, such as the possibility of asphyxiation, frostbite, and pressure problems, are associated with the use of cryogenic gases. To reduce the possibility of mishaps and guarantee the secure functioning of cryomilling machinery, adequate training and safety measures are important.
4. **Material Characteristics:** Spices vary in their physical characteristics and sensitivity to cryogenic temperatures, and not all of them are good candidates for cryomilling. The quality of the result may be impacted by certain spices being overly brittle or changing unintentionally in terms of texture, flavor, or scent during cryomilling.
5. **Environmental Impact:** Energy use and greenhouse gas emissions are two potential environmental effects of producing and using cryogenic gases, such as liquid nitrogen. Sustainability depends on efforts to reduce the environmental impact of cryomilling processes, such as through energy-efficient technology implementation and gas utilization optimization.

6. **Regulatory Compliance:** Ensuring adherence to regulations governing the processing, storage, and management of cryogenic gases in the food industry is crucial. The need to comply with standards related to food safety, hygiene, and quality control introduces additional challenges and administrative responsibilities to cryomilling processes.

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## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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