



# Effect of grinding temperatures on particle and physicochemical characteristics of black pepper powder



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## ABSTRACT

The cryogenic grinding method is extensively used to get the best quality of spice powders. The particle and physico-chemical characteristics of the ground black pepper powders produced at the grinding temperatures of  $-120$ ,  $-80$ ,  $-40$ ,  $0$  and  $40$  °C were investigated. The flowability and particle size demonstrated a positive relationship with the increasing grinding temperature for the powders, whereas, specific surface area and surface roughness of the particles showed a negative correlation. SEM analysis revealed the surface morphology and shape of the particles. The availability of mineral composition of the powder significantly increased with the reduction in the grinding temperature. Interestingly, the highest value was noticed for K followed by Ca and Cl at all the temperatures. Furthermore, a significant increase in the moisture content (10.5%), water activity (19.1%) and volatile oil content (20.1%) of the powder was noticed with the decrease in the grinding temperature. While the pH value and specific energy consumption for conveying and grinding were significantly decreased by 3.7 and 87.2%, respectively. Also, the total color difference of the powder was minimal at a grinding temperature of  $-40$  °C. However, no monotonous trend was observed for the gross calorific value of the powders.

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## 1. Introduction

Black pepper (*Piper nigrum* L.) is indigenous to India and is considered as “The King of the Spices” in the Food Industry [1–5] owing to its excellent quality characteristics like color, pungent taste, and aroma. Black pepper is principally utilized for flavoring and preserving foods as well as for pharmaceuticals, insecticide and cosmetic purposes [5–8], hence, it is most extensively used among the spices. A large quantity of spices produced industrially is in the form of powder. In the spice industry, powders derived by grinding of black pepper are mostly used as a valuable raw material for numerous food applications. Black pepper powder is usually preferred over the liquid form (oleoresins and essential oil) when used as a flavoring agent to be incorporated into the dry spice powder mixes (curry powder), beverage mixes, and dishes (cooked or uncooked).

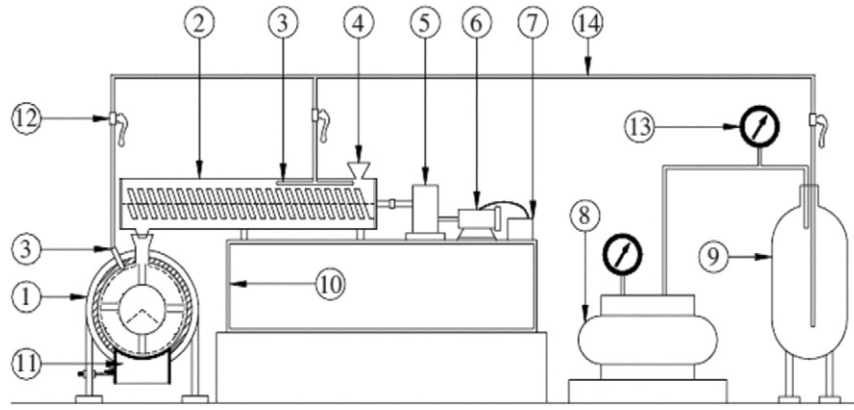
The size reduction of the spices assists in mixing of ingredients, transportation, and storage as well as enhances the availability of flavoring components and nutrients because of increase in the total surface area. Grinding is extensively used in different industries: food, chemical, agriculture, mining, and pharmaceuticals to obtain the powder. Grinding of spices is a complex process and can be defined as the characteristics size reduction operation practiced to obtain the spice powder. However, spices are difficult to grind due to their moisture,

volatile oil, fats, and fiber that hinders the particle size to be reduced below  $100\ \mu\text{m}$  in few minutes [9,10]. Moreover, a higher amount of mechanical forces is required to achieve the smaller particle size, resulting in higher energy requirement and heat generation that is harmful to the flavoring, nutritional and medicinal properties of the spices [11–14].

The hurdles of the conventional grinding process for grinding the spices can be ruled out by the promising cryogenic grinding (cryo-grinding) technology. The cryo-grinding of spices is a low temperature grinding process that can be defined as grinding of the spices with the aid of cryogen (cryogenic fluid) like liquid nitrogen ( $\text{LN}_2$ ), liquid carbon dioxide, etc. [11] to obtain high quality of spices powder. Many studies on the cryo-grinding of the spices have been investigated in the recent past that showed improvement in its volatile oil content or its important active flavoring component (black pepper: Piperine), color (better), particle size (fineness below  $5\ \mu\text{m}$ ), particle size distribution (uniform), total surface area (high), specific energy consumption (less), throughput (high) as well as no risk of fire, microbial load and air pollution as compared to conventional grinding process [1,11,15]. A few studies have been reported for cryo-grinding of black pepper [1,4,16] that has focused on the particle size and sensory attributes of the black pepper powder with a little importance on the fundamental knowledge about the powder characteristics that are required to understand the complex particle engineering. The cryogenic and ambient grinding of fenugreek seeds was reported to influence the specific energy requirement for grinding and flowability of the powders [17,18] significantly. Further, the particle size of the superfine ginger powder was reported to have

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**Fig. 1.** Cryogenic grinder set-up used for grinding of black pepper seeds. LEGEND: 1. Cryogenic grinder (Hammer mill); 2. Pre-cooler; 3. Liquid nitrogen distributor; 4. Feed hopper; 5. Reduction gear box; 6. Motor; 7. Rectifier; 8. Compressor; 9. Liquid nitrogen dewar; 10. Supporting frame; 11. Discharge hopper; 12. Flow control valve; 13. Pressure gauge; 14. Liquid nitrogen transfer line.

Source: Ghodki and Goswami, 2015 [10].

a significant effect on the physical-chemical properties of the powder [19].

The powder characteristics are affected by numerous variables like (a) material properties (intrinsic variables): moisture content, shape, size, etc., (b) milling/instrumental variables (extrinsic variables): rotor speed, feed rate etc., (c) environmental variables: relative humidity, temperature etc. of surrounding atmosphere which influence the intrinsic variables. Accordingly, significant diversity in powder exists. The effect of the significant environmental and intrinsic variables like moisture content, fat content, humidity, particle size distribution (PSD), temperature, particle shape on the powder characteristics have been revealed by numerous researchers [20–30] due to their growing importance in the food industry. Also, the specific energy requirement, physicochemical and flow characteristics of powder as well as the cost of machine and operation become the primary criteria for selection of an appropriate grinder.

Determination of powder flowability is crucial to prevent serious problems like agglomeration, sticking, etc. during production, storage, and transportation processes. The flowability of the pharmaceutical and food powders could be evaluated by different indicators like Hausner ratio (HR), static angle of repose ( $\alpha$ ), Carr index, flow index, slide angle, angle of internal friction, tensile strength, pouring time through a standard funnels and flow function coefficient value with precedent of their implementation may be found in generous works [22,23, 28,30]. HR and  $\alpha$  being easy to measure, considered as a useful indicator of flowability of the powders, and hence are widely used in the industry to distinguish the flow [21,23,25,28,31].

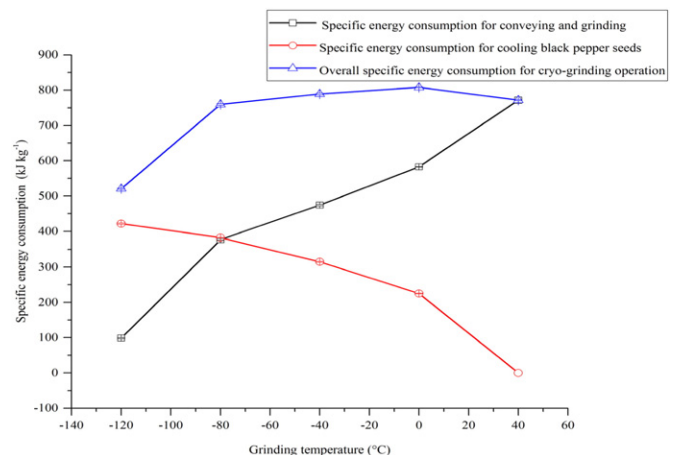
It is desirable to have the spice powders with the excellent sensory, safety and nutritional attributes such as color, texture, aroma, water activity, pH, minerals, etc. as well as adequate energy value which should be in agreement with the consumer and economic perspectives. Moreover, the color of spice powders is one of the crucial sensory attributes, which is influenced by many factors during grinding [11]. Moisture content and water activity play a significant role in determining the shelf stability of food [32,33]. Also, spices are considered to be a good dietary source of the minerals that may help to prevent the diseases if consumed in the required amount [34]. The inductively coupled plasma atomic emission spectrometry (ICP-AES) and Fourier transform infrared (FTIR) spectroscopy technique can be used for estimation of minerals but requires rigorous sample preparation technique, and also has lower detection limits as compared to scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) technique [35, 36] which is a qualitative and quantitative microanalysis method. SEM-EDX analysis has been reported by many researchers to characterize mango peel waste, concrete, fly ash, clay, cement, and zeolites [36, 37].

The quantitative investigation and comprehensive theoretical understanding of the powder (spices) properties influenced by the parameters of the grinding process are still deficient. Therefore, knowledge of the properties of black pepper powder such as flowability, particle size, morphology, moisture, water activity, etc. will aid in cost, capacity and process optimization of transportation, storage, and grinding operations as well as helps in better understanding of the mechanisms involved in this processes. Moreover, findings of the present study can be immensely utilized for the design of equipment for feeding, conveying, processing, handling, mixing, packaging, storing and transporting of the powder. Additionally, it facilitates the control over black pepper powder characteristics. The objective of this research is to investigate the influence of five grinding temperatures ( $-120$ ,  $-80$ ,  $-40$ ,  $0$ ,  $40$  °C) on the particle size, PSD, particle morphology, flowability, mineral composition, water activity, moisture, pH, volatile oil, color and energy value of the black pepper powder as well as to examine the energy required to obtain ground powder.

## 2. Materials and methods

### 2.1. Raw material

Black pepper (variety *panniyur-1*) seeds were provided by Indian Institute of Spice Research, Marikunnu, Calicut, India. The samples were initially sieved to eliminate the chaff, dust and dirt particles. Then the seeds were cleaned manually by removing broken, foreign matter, split, and immature seeds. The black pepper seed of the geometric



**Fig. 2.** Influence of grinding temperatures on specific energy consumption.

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