OPTIMIZATION OF PROCESS PARAMETERS FOR CRYOGENIC GRINDING OF DRIED GINGER

by

BHAVYA FRANCIS

KERALA AGRICULTURAL UNIVERSITY

Department of Food and Agricultural Process Engineering

KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND
TECHNOLOGY, TAVANUR - 679573, MALAPPURAM
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BHAVYA FRANCIS
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KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND TECHNOLOGY, TAVANUR - 679573, MALAPPURAM

KERALA, INDIA

2016

DECLARATION

I, hereby declare that this thesis entitled “**Optimization of process parameters for cryogenic grinding of dried ginger**” is a *bonafide* record of research work done by me during the course of research and the thesis has not previously formed the basis for the award to me of any degree, diploma, associateship, fellowship or other similar title, of any other University or Society.

Tavanur **BHAVYA FRANCIS**

Date: **(2014-18-105)**

Dr. Santhi Mary Mathew

Professor & Head

Department of Food and Agricultural Process Engineering

Kelappaji College of Agricultural Engineering and Technology

Tavanur

CERTIFICATE

Certified that this thesis entitled **"Optimization of process parameters for cryogenic grinding of dried ginger"** is a record of research work done independently by **Bhavya Francis (2014-18-105)** under my guidance and supervision and that it has not previously formed the basis for the award of any degree, diploma, fellowship or associateship to her.

Tavanur	**Dr. Santhi Mary Mathew**
Date:	(Chairman, Advisory committee)

CERTIFICATE

We, the undersigned members of the advisory committee of **Ms. Bhavya Francis (2014-18-105)** a candidate for the degree of Master of Technology in Agricultural Engineering majoring in Agricultural Processing and Food Engineering agree that the thesis entitled **"Optimization of process parameters for cryogenic grinding of dried ginger"** may be submitted by **Ms. Bhavya Francis (2014-18-105)** in partial fulfillment of the requirement for the degree.

Dr. Santhi Mary Mathew

Professor and Head
Dept. of Food & Agricultural Process Engineering
K.C.A.E.T., Tavanur
(Chairman)

Dr. Prince M. V.

Professor
Dept. of FAPE
K.C.A.E.T, Tavanur
(Member)

Dr. Rajesh G.K.

Assistant Professor
Dept. of FAPE
K.C.A.E.T, Tavanur
(Member)

Dr. E. Jayashree

Principal Scientist
Department of Crop
Production and Post Harvest
Technology, IISR, Calicut
(Member)

Dr. Seeja Thomachan

Assistant Professor
Dept. of Home Science
KAU, Thrissur
(Member)

Dr. Ravindra Naik

Principal Scientist
CIAE, Coimbatore

EXTERNAL EXAMINER

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Bhavya Francis

Dedicated to
My Family and
Teachers

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SYMBOLS AND ABBREVIATIONS

% : Per cent
ºC : Degree Celsius
& : And
/ : Per
= : Equal to
± : Plus or minus
≈ : Approximate
a* : Greenness to redness
Al : Aluminum
Anon. : Anonymous
a_w : Water activity
b* Blueness to yellowness
cm : Centimetre
d.b : Dry basis
et al. : And others
etc. : Etcetera
Fig. : Figure
g : Gram
h : Hour (s)
ha : Hectare
i.e. : That is
kg : Kilogram
kW : KiloWatt
L* : Lightness
m : Metre
m.c : Moisture content
min : Minute (s)
ml : Milli litre
mm : Millimeter
nm : Nanometer
No. : Number
pH : Pouvoir hydrogène
rpm : Revolution per minute
Rs. : Rupees

Sl.	:	Serial
viz.,	:	Namely
w.b	:	Wet basis
wt.	:	Weight
μg	:	Micro gram
ANOVA	:	Analysis of variance
BOPP	:	Biaxially oriented polypropylene
CV	:	Coefficient of variation
F	:	Feed rate
FID	:	Flame ionization detector
GC	:	Gas chromatography
HDPE	:	High density polyethylene
HP	:	Horse power
IS	:	Indian standard
K.C.A.E.T	:	Kelappaji College of Agricultural Engineering and Technology
KAU	:	Kerala Agricultural University
LCD	:	Liquid crystal display
LDPE	:	Low-density polyethylene
LN_2	:	Liquid nitrogen
PP	:	Polypropylene
RH	:	Relative humidity
SD	:	Standard deviation
SS	:	Stainless steel
T	:	Treatment
UK	:	United kingdom
USA	:	United states of America

INTRODUCTION

CHAPTER I

INTRODUCTION

Spices draw international attention as they are just became an unavoidable ingredient in the food, cosmetic, pharmaceutical and other important industries. It is costly due to its higher demand and limited production. India is the well established name in the global spice trade as it produces high quality spice and spice products and exports it to the global market. The requirement or demand for spices is always increasing as it explores newer ways to make better use of it.

Out of the 109 varieties of spices listed by the International Organization for Standardization (ISO), India produces nearly 63 items. The different climatic conditions in India provide sufficient span for the cultivation of a number of spices. The total spice and spice product export from India exceeded 8,43,255 tonness during 2015-16 (Spices Board, 2016). Almost all Indian states produce spices, with a total area of 3.15 million hectares (Anon., 2015a).

The major spices of India are black pepper, cardamom, chillies, ginger and turmeric. They are grouped according to their large production and higher demand. There are different forms of spices available which includes whole spices, organic, spice mixes, curry powders, spice blends, freeze dried, oleoresins, extracts, essential oils, de-hydrated, spice in brine and other value added products.

Spices can be used either in fresh or dried form. Most of the spices are used in the latter form and also in a ground state. For the production of value added products like spice extracts converting the spice to a ground state is necessary. The process of powdering or grinding results in smaller particle size product and it enhances easy handling, reduction in volume, ease of uniform mixing, and maximum release of flavor and aroma. The grinding operation relies on size reduction equipments like mills or grinders. The common mills used in food applications are hammer mill, pin mill, attrition mill etc. The traditional spice grinding employs these grinders and performs operation at ambient conditions.

The process of grinding any spice is an important operation as it involves some reduction in quality in the final product. During grinding, the temperature of the mill as well as the product rises as a result of converting mechanical energy to heat energy when a material is being fractured. And it may reach as high as 42-95^{o}C (Pruthi and Misra, 1963). This temperature rise due to energy dissipation in the form of heat is unfavorable in the case of spices. The volatile components such as essential oils in spice are heat sensitive (volatile) in nature and it may evaporate when the temperature is exceeding a particular limit. It infers that atmost care must be taken during grinding of spices.

Apart from the reduction of quality by loss of heat sensitive components like evaporation of volatile oils, the temperature rise during grinding results in melting of fat content in spices which leads to sieve clogging, higher energy consumption, poor grinder performance etc. Alternative methods like circulation of cold water around the grinder during milling are in practice to prevent the enormous heat generation, but they are found inefficient and failed to reduce the temperature rise significantly (Singh and Goswami, 1999).

Lowering the temperature during grinding finds numerous benefits in terms of product quality and grinding characteristics. The loss of volatiles can be significantly reduced by introducing cryogens during grinding (Pruthi, 1980). Liquid nitrogen used as cryogen with a boiling point of -195.6^{o}C provides the sufficient refrigeration effect to cool the raw material prior to grinding and maintains a low temperature throughout the grinding process.

Cryogenics is a technology which uses fluids with very low boiling points to cool materials by direct contact. It can cool materials to subzero temperatures and makes them brittle. Cryogenic grinding employs this embrittlement of a material and grinds them at cryogenic temperatures so that a material crumbles easily with less power requirement. The material to be ground is precooled using cryogens and then it is send to grinding where cryogen is used to maintain temperature (Russo, 1976). The fat and oil content in the spice will be frozen and there is no evaporation and melting so that the sieve clogging can be avoided.

In addition to maintaining the low temperature throughout grinding, vaporization of the cryogen (liquid nitrogen) to the gaseous state creates an inert and dry atmosphere which gives further protection for spice quality. Employing cryogens in the grinding operation either for precooling the raw spice or for the maintenance of low temperature within the mill or for both prevents the loss of volatile oils and moisture thereby retaining most of the flavour strength as the raw material (Gopalakrishnan *et al.*, 1991).

Ginger (*Zingiber officinale Roscoe*) is a major spice in India and is one of the oldest known spices to the mankind. The plant belongs to the family *Zingiberaceae*, and the rhizome is used as spice in fresh or dried form (Pruthi, 1993). Ginger (fresh or dried) found to be useful in food as well as in medicine. The pharmaceutical applications of ginger are well proven since ancient times and used widely in traditional medicines like Ayurveda.

The production of ginger in India is 6,83,160 tonnes from an area of 1,38,200 hectares in 2013-14 with a productivity of 4.9 tonnes/ha and the export is 40,400 tonnes in the year 2015. Export of ginger contributes 4.5 percent by volume and earns 2.23 percent foreign money of the total spice export. Kerala produced 21,249 tonnes of ginger accounting 3.2% of the national production in the same year (Spices Board, 2015).

Ginger is the major ingredient in a number of products like ginger candy, ginger ale, ginger bread etc. and used in curry powders, masalas, pickles, sauces and confectionaries. The value added products from ginger includes its essential oil and oleoresin which possesses higher demand in the international market as the extract replaces the bulk (Zachariah, 2008).

In most of the ginger products or products using ginger or in medicinal applications, ginger is used either in ground state or in the form of extracts. Either ways the spice has to be ground or convert to powder form for its end use. The dried ginger, used for grinding is too hard and very fibrous in nature. The problem with grinding of ginger is its fibrous nature. It makes ginger very difficult to grind and results in nonuniform distribution of particles in powder. But by imparting cryogenics in grinding it is possible to grind difficult spices.

A number of researches have been carried out on cryogenic grinding of different spices but not many studies on ginger. There is no much studies can that be found for the optimization of cryogenic grinding process for spices. Considering the advantages of cryogenic grinding, the present study was selected to grind dried ginger cryogenically to examine these advantages with the following objectives:

1) Study of physical and mechanical properties of dried ginger related to cryogenic grinding.
2) Optimize the grinding temperature for the maximum retention of quality.
3) Packaging studies of the ginger powder.

REVIEW OF LITERATURE

CHAPTER 2

REVIEW OF LITERATURE

One of the major aspects of food processing is to prevent or reduce the quality deterioration of the products as far as possible along with quantitative deterioration. The processing of each commodity or any agricultural material is designed with a focus of maximum retention of quality parameters. This chapter deals with the detailed review of research works carried out by various researchers related to the present study and also contains information about ginger and its benefits as a food ingredient.

2.1 Indian spices

India is known as the 'Home of spices' as it produces a wide variety and good quality spices. About sixty-three varieties of spices are cultivated in the country, which includes pepper (king of spices), cardamom (queen of spices), chillies, ginger, turmeric (5 major spices), coriander, cumin, cassia, celery and many others (minor spices) in its different agro climatic conditions (Pruthi, 1993).

Indian spices exports have been able to record strident gains in value. Spices exports have registered considerable growth in the World Spice Trade during the last five years. During 2015-16, a total of 8,43,255 tons of spices and its products worth Rs.16238.23 crore have been exported from the country as against 8,93,920 tons worth Rs.14899.68 crore in 2014-15, registering an increase of 9% in rupee terms of value (Spices Board, 2016).

2.2 Ginger -uses, varieties and market forms

Ginger has far wider applications as fresh ginger, dry ginger, ginger powder, oleoresin and ginger oil etc. in food processing, cosmetic and pharmaceutical industries. It is unavoidable in the manufacture of ginger bread, confectionaries, certain curried meats, ginger ale, curry powders, table sauces, in pickling and in the manufacture of certain cordials, carbonated drinks, ginger cocktail, liquors etc. The ginger oil is used as food flavourant in soft drinks (Anon., 2015b).

Ginger is the underground stem or rhizome of perennial tropical plant *Zingiber Officianale Roscoe* and being one among the 5 major spices of India (Pruthi, 1993). Ginger is cultivated in most of the countries like India, Jamaica, Nigeria, Southern China, Japan etc. Though there are other cultivators, Indian ginger (Cochin ginger) and Jamaican ginger are considered as the best in the world. It is an aromatic plant as a whole and the rhizome, fresh or dried is used as a spice. Ginger requires warm and humid climate and can be even grown in high altitudes up to 1500 meters above mean sea level.

There are several types of ginger available in the countries which are generally known by the localities where they are grown *viz,* Malabar ginger, Cochin ginger, Assamese ginger etc. The important varieties which are grown in different regions of the country are Nadia, Thingpui, thinglai, Maran, Wayanad Local, Rio-de-Janeiro, China, Suprabha, Ernad, Suruchi and Surabhi (Singh and Singh, 1996). Rajatha, Mahima and Varada are the improved varieties developed by IISR, Calicut. The market forms of dried ginger includes peeled, scraped or uncoated, rough scraped, unpeeled or coated, bleached, or limed clean peeled etc. according to the processing methods and requirements.

2.3 Health benefits of ginger

The medicinal properties of ginger are known to mankind since the time immemorial. It has been into the Ayurvedic medicine known as Mahaushadhi and is amongst the important herbs described. In addition, it has been used in traditional systems of medicine like Ayurveda, Chinese, Unani etc. for a wide range of ailments including pain, muscular aches, fever, sore throats, indigestion, vomiting, constipation, hypertension, dementia, fever, infectious diseases and helminthiasis (Ali *et al.,* 2008).

2.3.1 Anti microbial activity of ginger

Ginger is found to have direct anti-microbial activity and thus it can be used in treatment of bacterial infections (Tan and Vanitha, 2004). Guptha and Ravishankar (2005) reported ginger's strong antimicrobial activity against *Escherichia coli*. Ginger is found to be effective against several microbes like *Pseudomonas aruginosa, Staphylococcus aureus, Vibrio cholerae, Klebsiella spp.* and *Salmonella spp* (Islam *et al.,* 2014).

2.3.2. Anti diabetic effect of ginger

Mahluj *et al.* (2013) reported that ginger is a good remedy for diabetic patients to diminish the risk of some secondary chronic complications. From the clinical studies conducted on both humans and animals, ginger is proved to be anti diabetic due to the presence of several compounds in it (Al-Amin *et al.,* 2006). The studies strongly suggest that ginger is effective for the management of blood glucose levels and that the effects are both preventive and therapeutic for type-2 diabetes. The recent findings also suggest that ginger can directly increase glucose uptake in an insulin-independent manner that will be beneficial in the management of both type-1 and type-2 diabetes (Daily *et al.,* 2015).

2.3.3 Anti carcinogenic effect of ginger

Ginger has some anti cancer effects. The ginger extracts can be used to reduce the carcinogenic compounds of liver cancer and it can block some constituents from causing cancer in different parts of human body (Habib *et al.,* 2008). The chemo preventive property of ginger on oral carcinogenesis through suppression of tumor growth, proliferation of cells and induction of cell death has been reported by Khater

(2010). Also the gingerol content in ginger is found to be useful to defeat colon cancer (Ghosh *et al.*, 2011).

2.4 Structure and Composition of ginger rhizomes

The ginger rhizome is an irregularly shaped fibrous matter covered with a skin or peel and is consists of fingers. The main body is longish, somewhat flattened, and cylindrical with several primary and secondary branches (fingers) with narrowing pinkish tips. These fingers can be classified into primary secondary and tertiary (Jayashree and Visvanathan, 2011a). Cochin dried ginger is about 12 cm long and has a light-brown to yellowish-grey colour. The grading of rhizomes prior to export is done according to the number of fingers on the rhizomes.

The rhizomes generally contain moisture, protein, starch, carbohydrates, cellulose, fat, fibre and minerals along with a little steam volatile oil, pungent compounds and resins. The analysis of a market sample of fresh ginger resulted the following values: (as percentages) moisture: 80.9, protein: 2.3, fat: 0.9, carbohydrates: 12.3, fibre: 2.4, and minerals: 1.2. The principle minerals and vitamins in mg/100 g are Ca: 20, P: 60, and Fe: 2.6, the vitamins, thiamine: 0.06, riboflavin: 0.03, and ascorbic acid: 6.0. In addition to starch, the dominant carbohydrate the rhizome contains is 7.6% pentoses on a dry weight basis and small quantities of free sugars, glucose, fructose and sucrose (Govindarajan, 1982). The components present in ginger confirmed the usefulness of ginger root as a potential functional food and could be explored further in new product and formulation. The composition of ginger may vary with the agro-climatic conditions, variety, method of curing, drying etc.

The oval cross section (Fig. 1) shows a shriveled skin, an outer epiderm, an internal cork tissue, a cortex, an endoderm and a central cylinder. The oleoresin containing cells were marked as ‘ol’ in the figure. Both the cortex and central cylinder are dotted with fibrovascular bundles and oleoresin cells among the predominantly starch cells. The proximate composition of ginger is shown in Table 2.1.

Table 2.1 Proximate composition of cultivated varieties of ginger

Variety	Moisture (%)	Starch (by acid hydrolysis) (%)	Crude protein (N × 6.25) (%)	Crude fibre (%)	Ash		Water extract (%)	Acetone extract (%)	Volatile oil (%)
					Total (%)	Acid insol (%)			
Manjeri–	9.00	52.0	15.0	6.93	5.12	_	20.2	6.9	2.5
Wynad, local	10.00	50.7	13.7	8.20	6.33	0.02	19.7	6.8	1.9
Kunnamangalam	10.00	55.6	12.9	4.79	5.79	0.13	17.0	6.4	1.5
Maran	8.50	45.4	10.9	6.16	8.30	0.22	20.2	6.7	2.2
Nadia	11.50	59.0	10.5	5.67	8.23	0.11	20.1	3.9	1.0
Assam	10.00	40.4	10.3	9.70	7.50	0.03	22.4	9.3	2.4
China	9.60	52.8	11.8	8.00	9.28	0.28	15.5	5.8	1.4
Thingpuri	10.00	55.3	10.4	7.20	6.46	0.36	18.8	5.1	1.4
Rio de Janeiro	11.00	52.9	12.6	7.14	6.23	0.05	19.6	7.2	1.7

Source: Govindarajan (1982)

2.5 Ginger oil

The essential oil of ginger is volatile in nature and is obtained by stem distillation. The oil possesses the aromatic odour but not the pungent flavor of spice. Ginger yields 1-2.7% of essential oil. The colour of the oil varies from pale yellow to light amber and smell can be described as warm, but fresh woody and spicy (Zachariah, 2008). The essential oil of ginger is mainly comprised of 64.4% sesquiterpene hydrocarbons, 6.6% carbonyl compounds, 5.6% alcohols, 2.4% monoterpene hydrocarbons and 1.6% esters. The major compounds identified were Zingiberene and Sesquiphellandrene (Onyenekwe and Hashimoto, 1999). Table 2.2

shows the sesquiterpene hydrocarbons, oxygenated monoterpenes and monoterpene hydrocarbons present in the ginger oil. (Several sesquiterpene alcohols and other miscellaneous compounds are also identified but those are not given in table.) Table 2.3 shows the major compounds and their composition in the fresh and dried ginger oil.

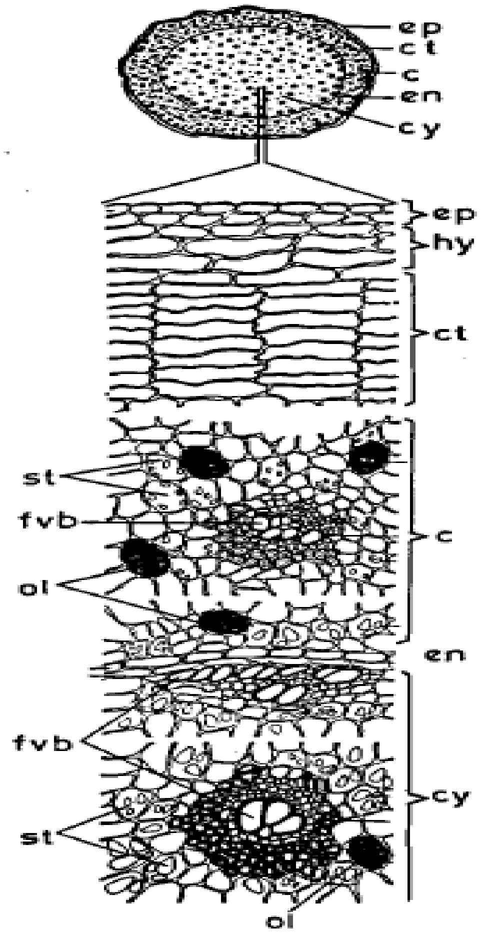


Fig. 2.1 Microscopic cross section of ginger rhizome (Oleoresin cells are marked as 'ol') (Source: Govindarajan, 1982)

The following tables show the different constituents identified in ginger oils (Table 2.2) and the composition of major compounds in fresh and dried ginger oils (Table 2.3).

Table 2.2 Constituents identified in ginger oils

Sesquiterpene hydrocarbons	Oxygenated monoterpenes	Monoterpene hydrocarbons
(−)-α-Zingiberene	*d*-Borneol	*d*-Camphene
β-Zingiberene	Bornyl acetate	Δ-3-Carene
(+)-*ar*-Curcumene	1:8 Cineol	*p*-Cymene
(−)-β-Bisabolene	Citrals a & b	Cumene
β-Elemene	Citronellyl acetate	*d*-Limonene
β-Farnesene	Geraniol	Myrcene
γ-Selinene	Linalool	*d*-β-Phellandrene
(−)-β-Sesquiphellandrene	α-Terpineol	α-Pinene
Sesquithujene		β-Pinene
		Sabinene

Source: Zachariah, 2008

Table 2.3 Composition of major compounds in fresh and dried ginger oils

Major compound	Fresh ginger oil (%)	Dried ginger oil (%)
Zingiberene	28.6	30.3
ar-curcumene	5.6	11
β-bisabolene	5.8	7.2
β-sesquiphellandrene	2.5	6.6
Geranial	8	4.4

Source: Sasidharan and Menon, 2010

2.6 Ginger oleoresin

Ginger oleoresin or the non volatile solvent extract is a value added product obtained from powdered dried ginger. It composed of the pungent principles of the dried spice and is dark golden brown viscous oil. Oleoresin is the spice extract which contains all the flavour components of the material contributing to aroma, taste, pungency and related sensory factors (Zachariah, 2008). Good ginger oleoresin contains 20–25% volatile oil; 25–30% pungency stimuli and the rest are non-flavour compounds such as fats, waxes and carbohydrates. The major compounds present are gingerols, zingerone and shagaols (Pruthi, 1999).

2.7 Engineering properties of agricultural materials

The knowledge about different properties of product is essential for the proper designing of processing equipments. The lack of knowledge may leads to time loss, energy loss and product loss. The engineering properties include physical properties mechanical properties, thermal properties, frictional properties etc. Different researchers made investigations to find the various properties of ginger as well as other similar materials. It is the fact that the properties of materials will vary accordingly. Here the works on various engineering properties of ginger and other similar materials are reviewed.

2.7.1 Physical properties

The physical properties of an agricultural material include size, shape, surface area, volume, density, porosity, colour and appearance (Sahay and Singh, 2001). Pandian *et al.* (2013) investigated the various physical and engineering properties of tamarind fruit such as average length, width, thickness, geometric mean diameter, sphericity index, surface area, bulk density, true density and porosity by adopting standard methods.

Jayashree and Visvanathan (2011a) studied the physical properties like average length, width and thickness of rhizomes, cylindricity of primary finger, mass, volume and surface area, bulk density, true density and porosity of fresh as well as dried ginger. The values are found to be 9.74 cm, 5.56 cm and 4.49 cm, 0.48, 31.62 g, 22.10 cm^3 and 65.84 cm, 460.09 kg m^{-3}, 1013.22 kg m^{-3} and 54.09% for dried ginger respectively.

2.7.2 Mechanical properties

Mechanical properties can be defined as the properties which affect the behavior of a material under applied force. The properties of agricultural materials like hardness, impact, compressive strength and shear resistance affect the processing operations and are useful for the designing of milling equipments. The properties like peel penetration force, compressive force at rupture and cutting force required penetrating through peel of rhizomes are studied in detail by Jayashree and Visvanathan (2011b).

2.8 Principles of grinding

The grinding is a reduction mechanism which comprises of deforming the material until it gets to the required particle size. It may be achieved by applying various forces. The types of forces commonly used in size reduction in food processing applications are compressive, impact, shear or attrition and cutting. In a grinding operation more than one force or a combination of forces are acting. The mechanical resistance offered by the body under the applied force describes all the properties that describe its behavior as it deforms and break (Loncin and Merson, 1979).

During the grinding process the material is stressed by the action of mechanical members of the grinding machine and initially the stress is absorbed internally by the material as strain energy. When the local strain energy, which is a function of the material, exceeds a critical level, fracture occurs along the lines of weakness and the stored energy is released.

Cracking behaviour of a material depends on whether the material is brittle or ductile, and on the type of stress applied. Grinding is therefore achieved by mechanical stress followed by rupture and the energy required depends upon hardness of the material and also upon the tendency of the material to crack (Cleef, 1991).

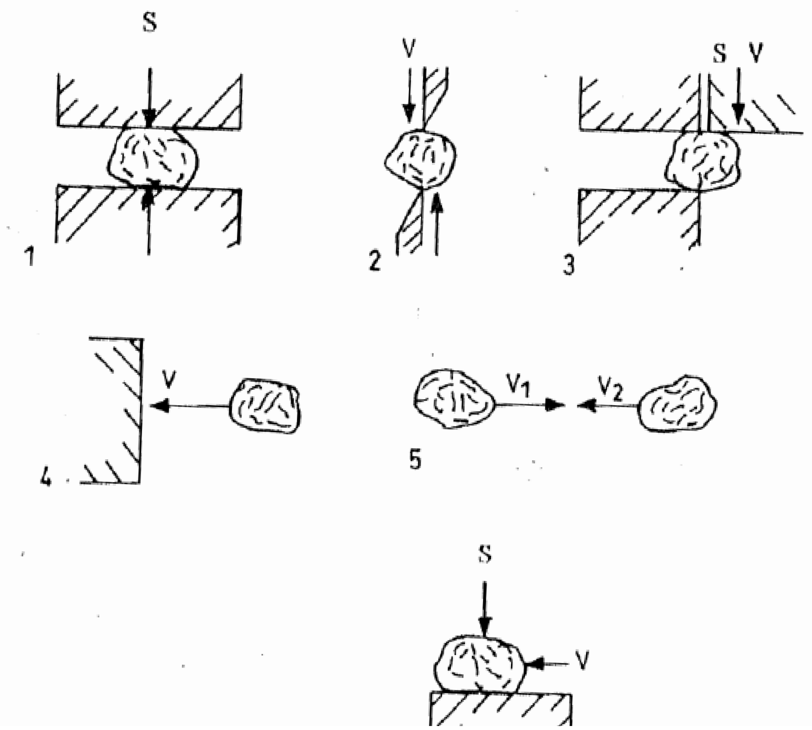
Table 2.4 Size reduction equipments used in food processing applications

Range of reduction	Generic name of equipment	Type of equipment
Coarse and intermediate	Crushers	Crushing rolls
Intermediate and fine	Grinders	Hammer mills Disc attrition mills, rod mills
Fine and ultra-fine	Ultra fine grinders	Hammer mills, tumbling mills (ball mills), pin mills

Source : Canovas *et al.,* 2005

2.8.1 Energy for grinding

According to Loncin and Merson (1979), the process of grinding requires a lot of energy and the 99% of the applied mechanical energy dissipated as heat. This energy dissipation results in rise in temperature of product and the equipment. The remaining 1% of energy is used for creating new surfaces and loosening the bond between particles. There is no general method for predicting the energy needed for size reduction. The energy needed for size reduction is depended upon elastic and plastic behavior of food material. These properties will vary with the distribution of moisture within the material and the rate at which stress applied.



1. Compression 2. Cutting 3. Shear 4. Impact 5. Impact 6. Rubbing

Fig. 2.2 Stress mechanisms in size reduction

2.8.2 Energy laws

2.8.2.1 Kick's Law

Kick’s proposed the law based on stress analysis of plastic deformation within the elastic limit. Kick’s assumed that the energy requirement for size reduction is a function of a common dimension of the material.

$$E = C \ln (D_1/D_2) \qquad \ldots\ldots(2.1)$$

Where,

E = Grinding energy in kJ

D_1 = Particle size of the feed in mm

D_2 = Particle size of the product in mm

C = Constant

2.8.2.2 Rittinger's Law

Rittinger assumed that size reduction is essentially a shearing procedure. Therefore the energy required is proportional to the new surface created, which in turn is proportional to the square of a common linear dimension.

$$E = C \left[\frac{1}{D_2} - \frac{1}{D_1}\right] \qquad \text{…..(2.2)}$$

Where,

E = Grinding energy in kJ

D_1= Particle size of the feed in mm

D_2= Particle size of the product in mm

C= Constant

2.8.2.3 Bond's Law

Bond reported a method for estimating the power required for grinding operation. According to this law the work required to form particles of size D_2 from very large feed is proportional to the square root of the surface to volume ratio of the product.

$$\frac{E}{W_i} = [100/D_2]^{(1/2)} - [100/D_1]^{(1/2)} \qquad \text{…..(2.3)}$$

Where,

D_1 and D_2 are the initial and final characteristic size of particle.

C= Constant

Wi = Work index, kWh/tone of feed

E = Grinding energy, kJ

2.9 Grinding of Spices

Grinding is one of the major unit operations performed on spices for its convenient use and for production of value added products. Spice grinding is an age-old technique just like any other food material. Additional care must be taken during grinding because the heat during grinding might lead to evaporation of volatiles and loss of aroma, results an inferior quality powder. There are a number of mills used to grind spices, and they are specially designed to cut, crush, or shatter the spice particle. The process of grinding ruptures a number of glands in the spices that contains oil and frees this oil for reaction or evaporation.

During grinding the applied mechanical energy converts into heat energy and it lead to the temperature rise in the mill. This temperature rise may be in the range 42-95°C (Pruthi and Misra, 1963).

Landwehr and Pahl (1986) reported a decrease in volatile oil content of pepper with increasing grinding temperature from -10°C to 50°C.

Grinding is an important operation in spice processing and requires a lot of care due to the possibility of quality deterioration. It is reported that a significant amount of volatile oil is lost during grinding due to the increase in temperature (Gopalkrishnan *et al.*, 1991).

Mathew (1998) reported that water circulation methods are in use to minimize the temperature during the grinding and it is achieved by modifying the grinder with water cooling jackets. But it resulted in poor heat transfer efficiencies and formation of cakes in the products.

The fat content in the spices also possess problems as it melts during grinding leads to the sieve clogging and further difficulties (Singh and Goswami,1999).

Gumming of cylinder walls and sieves are observed in large scale spice grinding units, due to continuous operation. Thus frequent stopping is needed to clean the mill causing energy loss and reduction in production capacity (Murthy, 2001).

Murthy and Bhattacharya (2008) reported that heat generation is unfavorable in the case of spices as it leads to the loss of volatile oils and other heat sensitive components resulting quality deterioration with reference to black pepper.

In case of ambient grinding, the specific energy consumption was found to be high because at high temperature (40 to 90^{o}C) oil comes out of cells and makes the product viscous and sticky in nature. This leads to high consumption of power in ambient grinding (Meghwal and Goswami, 2010).

The researches has been continued to maximize the quality parameters in the ground spice product just like the raw spice by various investigators. Pruthi, (1980) reported that the loss of volatile oil can be significantly reduced by adopting cryogenic technique in grinding.

Singh and Goswami (1999) reported that by using cryogen (liquid nitrogen at -195.6°C), it is possible to maintain the temperature at a desired level by absorbing the heat generated during the grinding operation, as it provides enough refrigeration required.

2.10 Cryogenics

Cryogenics is the branch of physics and engineering deals with the study of production of very low temperatures and the behavior of materials at these temperatures. The research about cryogens and cryogenics started in early 1840's at the time of Michael Faraday and now it has got so many applications in industry, defense and space programs. According to the international institute of refrigeration the cryogenic temperatures ranges from -153°C and below. The cryogenic conditions are produced using fluids known as cryogens.

2.10.1 Cryogenic fluids

Cryogenic fluids are fluids which boil at cryogenic temperatures and atmospheric pressure. Generally the fluids which boil at below room temperatures are termed cryogens (Edeskuty and Stewart, 1996). Liquid N_2, O_2, He, Kr, CH_4, CO_2 etc. exhibits the properties of cryogens. These fluids are used in different fields as per their properties, requirement, and system feasibility. Liquid nitrogen (LN_2) is the economical cryo refrigerant which offers more than 40 times more refrigeration capacity per unit volume than liquid helium and more than 3 times that of liquid hydrogen.

Table 2.5 Selected properties of food grade cryogens

Properties of cryogens	LN_2	CO_2 (Solid)
Density (kg m^{-3})	784	464
Boiling point (^{0}C)	-196	-78.5
Thermal conductivity (W m^{-1} K^{-1})	0.29	0.19
Specific heat capacity (liquid, kJ kg^{-1} K^{-1})	1.04	2.26

Latent heat of evaporation (kJ kg^{-1})	358	352

2.10.2 Theory of Cryogenic grinding

Cryogenic grinding involves grinding a material at cryogenic temperatures i.e. below ambient temperature. The heart of this technology is that it employs a cryogenic process to embrittle the material before grinding and then it can easily be fractured using mechanical force (Saxena and Soni, 2013).

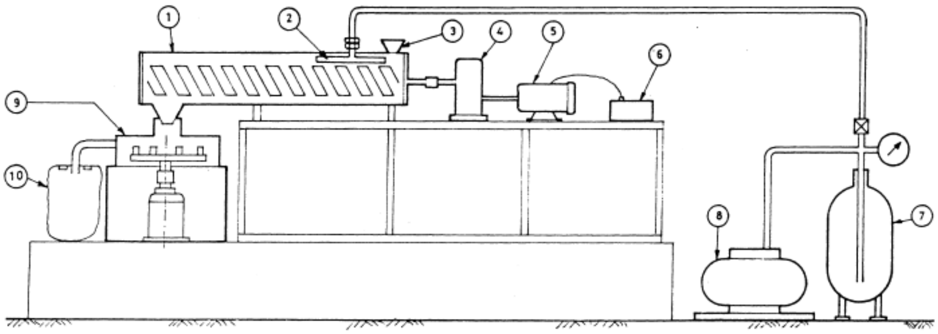
When a material comes in contact with a cryogen sudden heat transfer takes place and it will cause some internal stresses to build up due to fast freezing rate in such food materials during freezing. This internal stress may lead to cracking or shattering of frozen material (Kim and Hung, 1994).

The cooling of a material to very low temperature restricts the molecule flexibility in that material. Hence the stretching under the applied stress is reduced and the stress cannot be reduced by stretching any more. Thus a ductile material turns to be brittle under cooling. The cooling further affects the molecule movements so that a plastic deformation by viscous flows is reduced additionally. Altogether, this leads to an increasing probability of a fracture process (Wilczek *et al.*, 2004).

2.11 Cryogenic grinding of spices

Singh and Goswami (1999) proposed a cryogenic grinding system for spices. The system includes a precooler and a grinder. The precooler consists of a screw conveyor, a compressor, cryogen storage and a power transmission unit. The cryogen used is the liquid nitrogen with boiling point -195°C. The cryogen is used either to precool the raw material or to cool the grinding zone or for both. The liquid nitrogen comes in direct contact with the spice and evaporates as it cools the material leaving an inert atmosphere around the product. The grinding is carried out at predetermined temperatures using hammer mill, pin mill or attrition mill.

The extremely low temperature in the grinder solidifies oils so that the spices become brittle, they crumble easily permitting grinding to a finer and more consistent size (Saxena and Soni, 2013).



1. Screw conveyor 2. LN2 distributor 3. Hopper 4. Reduction gear box
5. Motor 6.Rectifier 7.LN2 Cylinder 8.Compressor 9. Grinder 10.Collecting bag

Fig. 2.3 Schematic view of cryogenic grinding system

Gopalakrishnan *et al.* (1991) have worked on cardamom and reported that cryogenic grinding using dry ice and liquid nitrogen in the centrifugal mill provided a superior quality product by preventing the loss of high volatile flavor constituents during grinding of cardamom.

Singh and Goswami (1999) found no further increase in volatile oil content in cumin seeds as the temperature decreased from -70 to -160°C. The mean diameter of particle and the specific energy consumption were increased as the temperature increased from -160 to -70°C. It is also observed that the grinding operation was smoother at temperatures lower than the brittle point of cumin seed and freezing point of cumin oil, as the oil got solidified.

Studies on cryogenic grinding of clove were conducted by Singh and Goswami (2000). They observed the influence of parameters, viz., grinding temperature, rotor speed, sieve size and feed rate on volatile oil content, specific energy consumption, particle size distribution, volume mean diameter, and sieve choking characteristics. It was observed that the clove could be successfully ground at temperatures below -50^{o}C without any deposition over the sieve surface. It is also observed that an increase in temperature in the cryogenic range (-110 to -50^{o}C) had no significant effect on volatile oil content, whereas temperatures in the range of 55 to 85^{o}C significantly reduced the volatile oil content from 11.0 to 9.3 mL/I00g. Thus, cryogenic grinding resulted in 29.5% more volatile oil in comparison to that of ambient grinding.

The effect of temperature of feed on grinding of black pepper (*Piper nigrum*) in a hammer mill using dry ice was studied by Mathew and Sreenarayanan (2007). They reported that the volatile oil content of the product showed 17% more retention of oil for ultra low temperature compared to the ambient condition (control). Gas chromatographic analysis of the essential oil showed higher retention of monoterpenes under ultra low temperature compared to ambient and low temperature conditions of grinding.

Murthy and Bhattacharya (2008) observed 50% more retention of volatile and a significant increase in monoterpenes in cryoground samples than the ambient ground. They optimized the cryogenic grinding conditions for maximum volatile oil content and a reasonable quantity of monoterpenes as 47 to 57 kg/h of feed rate, and -20 to -15^{o}C of product temperature.

From the observations of Meghwal and Goswami (2010), it is found that less specific energy and power were required for cryogenic grinding. Comparative study had shown that ambient grinding need more power (8.92%) and specific energy (14.5%) than cryogenic grinding. Particle size analysis of cryogenic grinding had shown that it produced coarser particles. The estimated requirement of LN_2 varied between 1 to 1.4 kg/kg for grinding temperature of -60 to -110^oC. It is also reported that cryogenic grinding is free from eye burning, sneezing and nose watering and it is a hygienically novel technique.

Saxena *et al.* (2013) studied the effect of cryogenic grinding on recovery of diosgenin content in fenugreek (*Trigonella foenum- graecum* L.) genotypes. In non cryo samples the diosgenin content was ranging from 1.3 to 1.5% and it is increased significantly in cryo ground samples and ranging from 2.1 to 2.5%. The cryogenic grinding technology was found superior in recovery of more diosgenin content from fenugreek seeds.

The effect of cryogenic grinding on volatile oil, oleoresin, total phenolics, and flavonoid content cumin seeds (*Cuminum cyminum L.*) have been analyzed by Sharma *et al.* (2014). It is found that cryogenic grinding helps in retaining the volatiles in ground product and enhanced its recovery. A significant increase in oleoresin percentage also observed in the cryoground samples.

Barnwal *et al.* (2014a) investigated the effect of cryogenic and ambient grinding on grinding characteristics of cinnamon and turmeric. The energy constants and specific energy consumption under cryogenic grinding were lower and the powder obtained had lower particle size.

In comparison with ambient grinding and cryogenic grinding of turmeric, the powder obtained with the latter is found to have good color and higher thermal conductivity (Barnwal *et al.*, 2014b).

The specific energy consumption, particle size and colour difference were found to be decreased as the grinding temperature (30 to -130^oC), rotor speed (2100 to 900 rpm) and feed rate were decreased in cryogenic grinding of cassia (Goswami and Ghodki, 2015).

Studies on effect of cryogenic grinding on volatile oil, total phenolics, oleoresin content, flavonoid content and anti-oxidant properties of seed extract of coriander (*Coriandrum sativum L.*) genotypes have been analyzed by Saxena *et al.* (2015). They observed a significantly high volatile oil and oleoresin content in cryogenically ground samples and concluded that cryogenic grinding technology is able to retain flavour and antioxidant properties of coriander irrespective of the genotypes.

Mallappa *et al.* (2015) conducted studies in chilli grinding using different grinding methods viz. Spice pulverizer, low temperature pulverizer, and cryogenic grinder. The capsaicin content and nutrients were found to be more in cryogenic grinder as compared low temperature pulverization and chilli pulverization. The colour value of chilli powder obtained from cryogenic grinder found much better than other grinding methods.

2.11.1 Advantages of cryogenic grinding

Murthy (2001) summarized the advantages of cryogenic grinding over ambient grinding. The salient features are as follows:

- Cryogrinding prevents the loss of volatiles and aroma during grinding to a great extent thus resulting superior quality spice powder. Spices retain their original flavor strength even after grinding.
- Cryogrinding reduces the oxidation of spice oils as the liquid nitrogen evaporates and creating an inert atmosphere in the grinding zone.
- It allows extremely fine grinding as the spice become brittle and the oil content solidify at very low temperature. This finely ground powder will have good dispersibility thus virtually eliminates specking problems.
- Cryogrinding makes it possible to grind difficult spices such as ginger as it causes fibres to shatter easily. It enables the grinding of high oil content spices such as nutmeg.
- Gumming up of grinding surfaces and screens and the sieve clogging can be reduced because the oil content is solidified and no melting fat.
- The mills performance as well as the throughput is increased as there is no heating and frequent stopping.

Table 2.6 Optimized conditions for different spices

Spice	Feed rate (kg/h)	Temperature (°C)	
Cumin	-	-70	Singh and Goswami (1999)
Pepper	-	-3.3 to -1.25	Mathew and Sreenarayanan (2007)
Black pepper	47–57	-20 to -15	Murthy and Bhattacharya (2008)
Coriander	1	below -50	Saxena and Soni (2013)
Cassia bark	2	-90.31	Goswami and Ghodki (2015)

2.12 Quality evaluation of ground product

2.12.1 Components in the volatile oil by GC analysis

Nampoothiri *et al.* (2012) studied and compared the essential oil constituents of three most popular cultivars of ginger in the sub Himalayan region. The volatile oils were isolated and analyzed through gas chromatography (GC) and gas chromatography-mass spectrometry. Eighty one components were identified and it is observed that zingeberene is the major compound found in all varieties.

Sasidharan and Menon (2010) compared the chemical composition of dried and fresh ginger oils by GC and GC-MS. It is reported that Zingiberene was the major compound in both ginger oils. Fresh ginger oil contained geranial (8.5%) as the second main compound and had more oxygenated compounds (29.2%) compared to dry ginger oil (14.4%). The dry ginger oil also contained ar-curcumene (11%), β-bisabolene (7.2%), sesquiphellandrene (6.6%) and δ-cadinene (3.5%).

2.12.2 Extraction of oleoresin

Nwaoha *et al.* (2013) studied the production of oleoresin from ginger peels and evaluated its antimicrobial and antioxidative properties. It also detailed the process of extraction of oleoresin from ginger by column percolation method using acetone as solvent.

2.13 Packaging of spices

In order to maintain the quality of the spices during handling, transportation, storage and distribution, the packaging material to be used is to be selected with care, keeping in mind the functional as well as the marketing requirements (Anon., 2012). The packaging requirements for spices, in general, are as follows:

- To protect the product from spillage and spoilage.
- To provide protection against atmospheric parameters such as light, heat, humidity and oxygen. The selected packaging materials should have high water vapour and oxygen barriers.
- The packaging material should have a high barrier property to prevent aroma/flavor losses and ingress of external odour.
- The volatile oil present in the spice product has a tendency to react with the inner/contact layer of the packaging material, at times leading to a greasy and messy package with smudging of the printed matter. The packaging material should therefore be grease and oil resistant and compatible with the product.
- Besides the above functional requirements, the packaging material should have good machinability, printability and it should be easily available and disposable.

The knowledge about the factors causing spoilage during storage will help to choose the packaging material/type of package for a specific product. The spoilage factors for spices are its moisture content, loss of aroma/flavour, discolouration, insect infestation and microbial contamination. Both rigid and flexible packages are used in whole spice and ground spice packaging. The packaging materials can be jute, glass, metal, paper boards, plastic etc. and the packaging articles can be gunny sacks, glass bottles, metallic tins, paper cartons, rigid plastic bottles/boxes, flexible plastic films/pouches etc.

The printed flexible pouches become very popular due to their easy availability, excellent printability, machinability, light weight and cost-effectiveness. They are generally laminates of various compositions. The commonly used laminates are: polyester/metallised polyester/LDPE, BOPP/LDPE, BOPP/metallised polyester/LDPE and polyester/Al foil/LDPE. The properties of LDPE includes it is heat sealable, chemically inert and have good moisture barrier qualities.

Sindhu *et al.* (2010) suggested that laminated aluminum foil and plastic jars are suitable for long duration storage of turmeric powder under refrigerated as well as ambient conditions followed by HDPE film. Storing powder in LDPE and PP causes stickiness in product. The study also observed that the storage condition has no significant effect, whereas storage duration and packaging material has significant effect on the quality of turmeric powder.

Meghwal and Goswami (2014) conducted studies on packaging of fenugreek and black pepper powder with different packaging materials *viz*, glass jar, steel jar, plastic jar, aluminum bag and poly bags and stored under normal atmospheric conditions. However, glass jar and steel jar were found to be better containers for storing powders for longer storage period than other options. Based on powder quality retention, the order of packaging materials may be as follows: Glass jar > Steel Jar > Aluminum bag > Plastic jar > Poly bag.

Adinoyi *et al.* (2015) studied the possible short term storage effect on some chemical properties of locally processed turmeric powder. LDPE, polypropylene and glass bottles were used and stored at room temperature. It is observed that the packaging materials maintained the quality parameters of powder in-terms of spectrophotometric absorbance and pH. It also recommends 4 weeks storage periods for turmeric powder.

MATERIALS AND METHODS

CHAPTER III

MATERIALS AND METHODS

The present chapter describes the details of materials and methods employed for determining various physical and mechanical properties as well as the grinding characteristics by ambient and cryogenic grinding of dried ginger.

3.1 Materials

Fresh ginger rhizomes (variety Malabar) were procured in bulk from a local farmer. After cleaning and partial peeling they were dried under sun for one week to attain storage moisture content. The moisture content of the rhizomes, after equilibration, was determined as per the method mentioned in section 3.2.1.

3.2 Study of physical and mechanical properties of dried ginger related to grinding

The details of the materials and methods followed for determining the various properties of dried ginger are described in this section. The properties to be studied are moisture content, shape, hardness, freezing point of volatile oil and crushing strength at different temperatures.

3.2.1 Moisture content

The moisture content of the dried ginger was determined using entrainment technique by co-distillation with toluene, according to (Pruthi, 1999). The equipment used was the Dean-Stark apparatus in which the amount of water is determined by trapping it to a graduated tube (Plate 3.1). Ten g of ground sample and 100 ml toluene were used for the analysis. The toluene was poured to the ground sample which is kept in a round bottom flask. The flask is attached to Dean-Stark apparatus and a condenser. The receiver trap of Dean-Stark apparatus should be filled with toluene before the experiment starts. On boiling the water content in the sample along with toluene gets evaporated, condensed and trapped into the receiver of the apparatus which already contains toluene. The distillation was continued till the volume of water became constant. The water content will be at the downside of the trap due to density difference. The apparatus is allowed to reach the room temperature until the separation line between toluene and water become clear. The reading was noted from the graduated tube. The moisture content was then calculated by the following equation:

$$\text{Moisture content (w.b.,\%)} = \frac{\text{Vol .of water (ml)}}{\text{Weight of sample (g)}} \text{x100} \qquad \text{…..(3.1)}$$

Plate 3.1 Dean-Stark apparatus

3.2.2 Shape of dried rhizomes

The shape of the rhizomes may be expressed in terms of sphericity and roundness (Kaleemullah and Kailappan, 2003). The sphericity shows the degree of closeness of a material to the shape of sphere. Higher the sphericity value, closer its shape to a sphere. Roundness measures the sharpness of the corners of the solid. The roundness and sphericity, of the ginger were calculated by tracing the magnified shadowgraphs of 10 gingers in three mutually perpendicular positions on a graph paper, with the help of a projector. The projected area, diameters of the largest inscribing and the smallest circumscribing circles of the projected view were measured. The roundness and sphericity were calculated by using the following equations:

$$r = \frac{A_p}{A_c} \qquad \text{…..(3.2)}$$

and

$$S = \frac{d_i}{d_c} \qquad \text{…..(3.3)}$$

Where,

R = Roundness, %

A_p = Largest projected area of the particle, mm^2

A_c = Area of smallest circumscribing circle, mm^2

S= Sphericity, %

d_i= Diameter of largest inscribing circle, mm

d_c = Diameter of smallest circumscribing circle, mm

3.2.3 Hardness

Hardness of a material is defined as the resistance to deformation under applied force. It is determined by performing a hardness test using hardness tester. The equipment used is the Rockwell hardness tester (Saroj RAB 250).The dried rhizome was placed horizontally upon the flat surface of the analyzer and the test load is applied. The results of 10 replications were recorded.

3.2.4 Freezing point of volatile oil

The freezing point of volatile oil is determined as given by Meghwal and Goswami (2011). The dried ginger oil was extracted by the steam distillation technique. Liquid nitrogen was used to determine the freezing point of the oil. Nearly 5 ml of oil was taken in a beaker and liquid nitrogen was directly poured into the oil. The oil was continuously stirred using a glass rod. The freezing point was obtained when solidification of oil started and it is indicated by a sudden temperature rise. The temperature of oil was recorded by a temperature sensor with a range of temperature of -200 to 100^{o}C with a least count of 0.1^{o}C.

3.2.5 Crushing strength at different temperatures

The crushing strength of ginger is determined by performing compression test using texture analyzer (Stable Micro Systems, UK). The instrument has a micro processor regulated texture analysis system interfaced to a personal computer. The instrument consists of two separate modules; the test-bed and the control console (keyboard). Both are linked by a cable which route low voltage signal and power through it. The texture analyzer measures force, distance and time and hence provide a three-dimensional product analysis. Forces may be measured to achieve set distances and distances may be measured to achieve set forces.

The dried rhizome was placed horizontally upon the flat surface of the analyzer and the probe is fixed. 50 kg load cell is used and the compression force was applied at a speed 0.5 mm/s. It is allowed to compress the material for 2 mm from the point of contact. Type of probe used is P-75 platen and the trigger force was 20 g.

The different test temperatures are attained by dipping the dried ginger into the liquid nitrogen. A thermocouple (measure from 100 to -200^{o}C) is used to monitor the temperature of the specimen. The test is done for all the test temperatures with 5 replications. The texture analyzer and the probe used are shown in the Plate 3.2.

Plate 3.2 Texture analyzer

3.3 Studies on effect of existing grinding method on quality of dried ginger powder (ambient grinding of dried ginger)

To study the effect of existing grinding method on quality of dried ginger powder, a grinding operation was performed at ambient conditions without any cooling or temperature controlling method. The grinding of dried ginger was done using a hammer mill with the following specifications: hammer mill (fixed type), 1.5 HP motor, 3 phase, 440 V, 1440 rpm. The grinder is modified with three temperature sensors (thermocouples) to indicate the temperatures of feed, grinding zone and product outlet regarding the present research work. The thermocouple at feed inlet (Creative DTI 308) is used to measure temperature of feed and can measure temperature ranging from 0 to 100^{o}C. The other two thermocouples are of the same kind (Selec TC513) with a measuring range -200 to 100^{o}C. The sensors are of bead type and one is inserted into the grinding zone and the other is fixed at the outlet giving the instantaneous temperature. The reading will be shown in the display box attached to it. A three phase energy meter (L&T ER300P) is attached to the grinder to measure the power requirement.

250 g of dried ginger was taken and the grinding operation was performed at ambient conditions. The time of grinding, energy usage and the temperatures at feed, grinding zone and product outlet were noted. The time taken was noted down by means of a stop watch. The experiment is conducted thrice and the powder is then collected, packed and sealed in a polythene cover immediately and stored at refrigerated condition. It is opened only at the time of analysis.

The obtained product was analyzed for its physico-chemical properties like particle size, volatile oil yield, components in volatile oil, oleoresin content and colour. It is also analyzed for functional properties like water activity, bulk density and flowability.

3.3.1 Particle size

The average particle size was determined by sieve analysis (Sahay and Singh, 2001). Indian standard sieves (IS sieves) are used with a mechanical sieve shaker. IS sieves 100,70,50,40,30,20 and 15 along with a pan were used with openings 1.00, 0.708, 0.500, 0.420, 0.296, 0.211, and 0.157 mm respectively. 50 g powder is used for analysis and the shaking time was 10 minutes. The material retained on each sieve is weighed using electronic balance with accuracy ±0.01g and is converted to percentage of total mass to determine fineness modulus. The fineness modulus (FM) is calculated using the following equation:

$$\text{FM} = \frac{\text{Total percent retained on sieve}}{100} \qquad \ldots\ldots(3.4)$$

And the average particle size is calculated from the fineness modulus as;

$$D_p = 0.135\,(1.366)^{FM} \qquad \text{......(3.5)}$$

Where, D_p = average particle size in mm

3.3.2 Volatile oil

Volatile oil of ginger powder was extracted by hydro-distillation method using Clevenger apparatus (Pruthi, 1999). The powder weighing 50 g was kept in a round bottom flask of capacity 1000 ml along with sufficient distilled water. It is allowed to distill for 5 hours or until there is no change in the oil yield. The oil will be trapped at the graduated portion of the Clevenger tube and the yield can be easily noted down. The Plate 3.3 shows the apparatus setup along with the condenser and heating mantle.

The percent oil content can be calculated as

$$\text{Volatile oil content (\%)} = \frac{\text{Volume of volatile oil (ml)}}{\text{Dry weight of sample (g)}} \text{ X } 100 \qquad \text{.....(3.6)}$$

Plate 3.3 Clevenger apparatus

3.3.3 Analysis of volatile oil

The analysis of volatile oil components in the dried ginger oil is performed using gas chromatographic (GC) technique (Sasidharan and Menon, 2010). (Model: SHIMADZU GC-17A). In gas chromatography, the solution to be analyzed (essential oil) is injected to into the instrument and there it enters to a gas stream (carrier gas). The carrier gas (mobile phase) transports the injected sample into a separation tube known as column. The sample is vaporized by the heated system and the components in the sample are separated inside the column. The detector measures the quantity of the components that exit the column. Standard sample with known concentration is required to inject into the instrument to measure sample with unknown concentration. The widely used detector is flame ionization detector (FID) having good sensitivity to almost all organic compounds. The major components like zingiberene, citral a (geranial) and citral b (neral) were analysed, and the citral standard was purchased from Sigma-Aldrich.

The column used was Thermo TR-1with specifications: length and thickness, 30m x 0.25 mm, and film thickness, 0.25µm. 1µl oil is used for each analysis. The conditions were as follows: Temperature programming: from 60^oC-200^oC, rate at 5^oC /min, hold at 60^oC for 5 min, hold at 200^oC for 10 min, FID temperature 280^oC, injection temperature 250^oC, carrier gas used : nitrogen at a flow rate of 1mL/min, split ratio of 1:30. The GC equipment is shown in Plate 3.4.

3.3.4 Ginger oleoresin

Cold extraction method was used to determine the oleoresin content in ginger. Nwaoha *et al*. (2013) detailed the process of extraction of oleoresin from ginger by column percolation method using acetone as solvent. 10 g sample was kept in a glass column percolator along with 100 ml acetone. It is allowed to stand for 24 hours and then the extract is collected. Again 30 ml acetone is added to the column and kept for

2 hours to collect the remaining oleoresin content. Both the extracts were pooled together and the solvent is removed using a rotary flash evaporator. The percentage oleoresin content of the ginger powder is calculated from the following expression,

Plate 3.4 Gas Chromatographic Equipment

$$\text{Oleoresin (\%)} = \frac{\text{weight of extracted material}}{\text{weight of sample}} \times 100 \qquad \text{.....(3.6)}$$

3.3.5 Colour of the powder

The colour of the ground ginger was measured using Hunter Lab colourimeter (Colorflex EZ, Hunter Associates laboratory Inc., Reston, Virginia, USA). The Hunter lab's colour flex spectro calorimeter (Plate 3.5) consists of measurement (sample) port, opaque cover and display unit. This colour flex meter works on the principle of focusing the light and measuring energy reflected from the sample across the entire visible spectrum. For matching a series of colour across the visible spectrum, primary lights are required and describes the colour by mathematical model called as Hunter model. It reads the colour of sample in terms of L*, a* and b* values where, luminance (L) forms the vertical axis, which indicates darkness to whiteness (0 to 100). Chromatic portion of the solids is defined by: a (+) redness, a (-) greenness, b (+) yellowness, and b (-) blueness.

A transparent glass cup filled with sample was placed over the port of the instrument and an opaque cover which act as a light trap to exclude the interference of external light was placed over the cup. Colour was calibrated by fixing the definite colours like white and black tiles. After calibration, the sample was placed over the port and values of L*, a* and b* were recorded.

3.3.6 Specific energy consumption

A three phase energy meter (L&T ER300P) was connected with the grinder to measure the power consumed and to measure the energy required in grinding (Singh and Goswami, 1999). The following expression is used to calculate the specific energy consumed in grinding:

$$\text{Specific energy consumption} = \frac{Power\ \ consumed\ \ (W)\,X\ \ 3.6}{Feed\ rate\ \ (kg/h)} \qquad \ldots\ldots(3.7)$$

3.3.7 Water activity

The water activity of ginger powder was carried out using Aqua lab water activity meter (M/s. Aqua Lab, U.S.A; model: Series 3TE). Water activity or a_w is the ratio of partial vapor pressure of water in a substance to the standard state partial vapor pressure of water. It determines the lower limit of available water for microbial growth in a product i.e. water activity is a critical factor that affects the shelf life of products. It predicts stability of a product with respect to microbial growth and rates of deteriorative action.

To determine the water activity, 3 g of powder was placed in the chamber in a disposable cup and the chamber is closed (cup with over-filled sample may contaminate the chamber‘s sensors). In the chamber, water activity is measured by equilibrating the liquid phase water in the sample with the vapor phase water in the headspace of closed chamber and measuring the relative humidity of the headspace. It uses a dew point sensor, temperature sensor and an infrared thermometer. The water activity values were noted down from the LCD display. Plate 3.6 shows the water activity meter used for the analysis.

Plate 3.5 Hunter Lab colorimeter

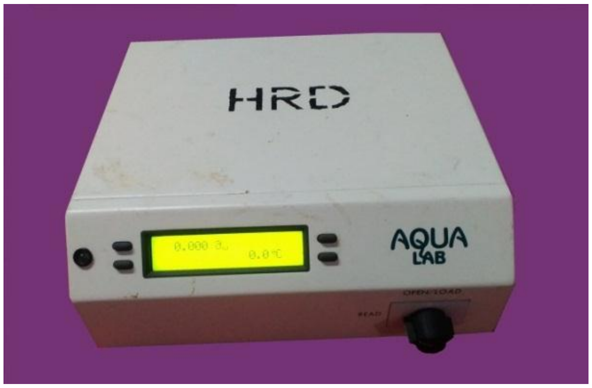


Plate 3.6 Water activity meter

3.3.8 Bulk density

Bulk density was determined by adding 10 g of ginger powder to a 50 ml graduated cylinder without any compaction (Phoung and Sertwasana, 2010). The bulk density value was calculated from mass of the powder and the volume occupied in the cylinder. It is calculated as:

$$\text{Bulk density (g/cm}^3\text{)} = \frac{Weight\ of\ powder}{Volume} \qquad \text{.....(3.8)}$$

3.3.9 Flowability

The flowability of the powder was measured in terms of angle of repose (Geldart *et al.*, 2012). The powder is allowed to fall from a funnel through a small opening and allow forming a conical pile on a horizontal surface. The funnel height should be maintained 2-4 cm from the top of the pile to minimize the impact of fall on the pile tip. The height of the pile and the radius of the base circle are measured. 10 g of powder was used and the height of fall is 10 cm. The angle of repose is calculated as

$$\Theta = \tan^{-1} \frac{h}{r} \qquad \text{…..(3.9)}$$

Where,

Θ= Angle of repose in degrees

h = Height of pile in cm

r = Radius of base circle of pile formed in cm.

The flow properties and corresponding angle of repose are given in Table 3.1

Table 3.1 Flow properties and corresponding angle of repose

Flow properties	Angle of repose (degree)
Excellent	25-30
Good	31-35
Fair	36-40
Passable	41-45
Poor	46-55
Very poor	56-65
Very very poor	>66

3.4. Cryogenic grinding of dried ginger

The ordinary hammer mill is converted to a cryogenic grinder by giving it a liquid nitrogen/cryogen supply. The additional requirements for this conversion are a cryogen storage tank (dewar) and a transferring valve with distribution tube.

3.4.1 Dewar

The liquid nitrogen is kept in a storage tank known as dewar. These are specialized types of vacuum flask used for storing cryogens. Dewars can be of different types including open buckets, flasks with loose-fitting stoppers or self-pressurising tanks. All dewars have walls constructed from two or more layers, with a sufficiently high vacuum maintained between the layers. This provides very good thermal insulation between the interior and exterior of the dewar, which reduces the rate of loss of cryogen to the surroundings.

3.4.2 Transfer valve and distribution tube

The liquid nitrogen is supplied to the grinder through an insulated tube which can be operated by a valve. The transfer tube must be designed for reduced product evaporation and elimination of frost and condensation during transfers. The combination of vacuum jacket and high insulation make this tube able to meet its functional requirements and makes it efficient flexible transfer line. The proper and efficient insulation reduces the loss of cryogen to atmosphere thus saving cost.

3.4.3 The grinding process

One end of the transfer tube is opened to the grinding zone and the other end is inserted to the dewar. When the valve is opened, the liquid nitrogen flows through the tube and reaches the grinding zone of the grinder directly and chills the area. The required temperature in the grinding zone can be attained and maintained by adjusting the valve. The liquid nitrogen supply should be given to the grinder before feeding starts, to attain the pre determined temperature. When the grinder attains the pre set temperature, the feed is given through the feed hopper and the grinding starts. The temperature of the grinding zone is continuously monitored by temperature sensors. In the case of temperature increase during grinding, the flow rate of liquid nitrogen was increased by increasing the opening of transfer line valve. The energy used, time of grinding and the temperatures of feed, grinding zone and product were recorded for each operation. The cryogenic grinder along with dewar and transfer valve are shown in Plate 3.7. The temperature display box (Plate 3.8) and the temperature sensors (probes) used (Plate 3.9) are also shown.

3.5 Experimental design

Based on the previous works and preliminary studies, temperature at the grinding zone and feed rate were fixed as independent variables. Two different feed rates and five different temperatures have been selected as per the experimental design. The quality parameters which are characteristic to these parameters were selected as dependent variables.

The treatments were as follows:

FITI: Grinding at feed rate 8 kg/h at grinding temperature -10^{o}C

FIT2: Grinding at feed rate 8 kg/h at grinding temperature -40^{o}C

FIT3: Grinding at feed rate 8 kg/h at grinding temperature -70^{o}C

FIT4: Grinding at feed rate 8 kg/h at grinding temperature -100^{o}C

FIT5: Grinding at feed rate 8 kg/h at grinding temperature -130^{o}C

F2T1: Grinding at feed rate 20 kg/h at grinding temperature -10^{o}C

F2T2: Grinding at feed rate 20 kg/h at grinding temperature -40^{o}C

F2T3: Grinding at feed rate 20 kg/h at grinding temperature -70^{o}C

Plate 3.7 Cryogenic grinder

Plate 3.8 Temperature display box

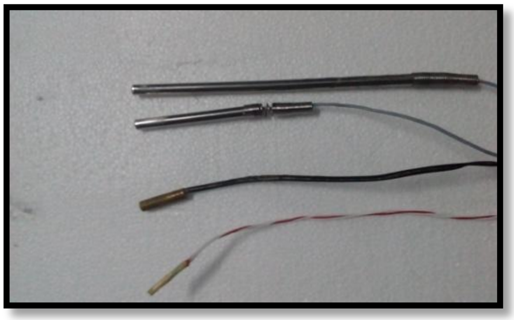


Plate 3.9 Temperature sensors

F2T4: Grinding at feed rate 20 kg/h at grinding temperature -100°C

F2T5: Grinding at feed rate 20 kg/h at grinding temperature -130°C

F3T1: Grinding at feed rate 35 kg/h at grinding temperature -10°C

F3T2: Grinding at feed rate 35 kg/h at grinding temperature -40°C

F3T3: Grinding at feed rate 35 kg/h at grinding temperature -70°C

F3T4: Grinding at feed rate 35 kg/h at grinding temperature -100°C

F3T5: Grinding at feed rate 35 kg/h at grinding temperature -130°C

3.5.1 Independent variables

a) Temperature of grinding
b) Feed rate

3.5.2 Dependent variables

I. Characteristics of grinding
a) Specific energy
b) Particle size
c) Time of grinding

II. Quality characteristics of ground material
    a) Moisture content of the powder
    b) Volatile oil
    c) Volatile oil components
    d) Oleoresin
    e) Colour of the powder

250 g of dried ginger was used in each trial. The ground product is collected, packed in polythene cover, sealed and kept in refrigerated conditions and opened at the time of analysis. The analysis of powder is carried out for the physico-chemical

properties such as particle size, volatile oil, volatile oil contents, oleoresin and colour of the powder, and for functional properties like water activity, bulk density and flowability. The procedure followed is the same as mentioned before.

3.6 Packaging and storage studies of the dried ginger powder

The powder obtained by the optimized process condition was packed for shelf life studies. The samples were packed in different packaging materials *viz*, LDPE 100 gauges, LDPE 200 gauges, LDPE 400 gauges and laminated aluminum foils. The packages are sealed using hand sealing machine and stored in ambient condition (temp 29-30^oC with 40-50% RH) for 3 months and the different quality parameters of the powder were evaluated in each month. The parameters checked are moisture content, volatile oil, oleoresin and colour of the powder. Functional properties like water activity, bulk density and flowability were also noticed. The procedure followed was the same as mentioned before.

3.7 Statistical analysis

The results were statistically analyzed to determine the effect of temperature and feed rate on different dependant variables. The software used was Design Expert (version 7.0.0). The optimization for grinding temperature and feed rate for the best quality ginger powder was done using the same software. The design followed was general factorial with 2 categorical factors *i.e.*, feed rate and temperature of grinding. 12 responses have been selected for the optimization viz. particle size, specific energy consumption, time of grinding, moisture content, volatile oil, volatile oil contents *i.e.*, zingiberene, geranial and neral, oleoresin, L*, a* and b* of the powder. Numerical optimization has been done with the following criteria: particle size- minimize, specific energy consumption - minimize, time of grinding- minimize, moisture content- minimize, volatile oil- maximize, volatile oil contents- maximize, oleoresin- maximize, L* - maximize, a*- minimize and b* -maximize. The treatment with the highest desirability value has been selected as the best method in terms of good grinding characteristics and product quality.

RESULTS AND DISCUSSION

CHAPTER IV

RESULTS AND DISCUSSION

The outcomes of the various experiments conducted to optimize the process parameters, various physical properties and grinding characteristics involved are enunciated and discussed in this chapter. The chapter has been subdivided as follows;

4.1 Physical and mechanical properties of dried ginger
4.2 Studies on existing grinding method of dried ginger
4.3 Cryogenic grinding of dried ginger
4.4 Packaging studies of ginger powder

4.1 Physical and mechanical properties of dried ginger

The knowledge about the engineering properties of agricultural products is always essential for the selection and design of processing and handling equipments. Here the study is restricted to some selected parameters which directly or indirectly affect the grinding operation. The various parameters were determined for dried ginger as per the methods explained in chapter 3.

4.1.1 Moisture content of raw material

The fresh ginger (variety: Malabar maran) was dried under sun until the moisture content reaches a safe level. The material dried for one week and the moisture content was checked using toluene distillation method. The moisture content of the dried ginger was found to be 10.6 % (w.b.).

4.1.2 Shape of ginger

The shape of ginger has been determined in terms of sphericity and roundness according to the methods explained in 3.2.2, and was found as 45.1% and 14.04% respectively. The shape of dried ginger was irregular.

4.1.3 Hardness

Hardness is the resistance of a material to deform under the applied force. The hardness of the ginger was found using a hardness tester as mentioned in section 3.2.3. Hardness of 25 samples was recorded and the mean value was taken and it was found to be 172.3 N.

4.1.4 Freezing point of volatile oil

The freezing point of essential oil of ginger was determined by direct cooling using liquid nitrogen (Meghwal and Goswami, 2011) and was found to be -46^{o}C. The method followed was described in the section 3.2.4. The data on the solidification point of essential oil is useful for adjusting the temperature for low temperature grinding of spices such as cryogenic grinding. The cryogenic grinding involves embrittlement of raw material and the solidification essential oil increases the degree of embrittlement. Grinding the material at solidification temperature helps in all the volatile oil and aroma content to be retained in the product.

4.1.5 Crushing strength of dried ginger at different temperatures

The crushing strength of the ginger was determined at different temperatures by texture analyzer (Stable Micro Systems, UK) as per the method cited in 3.2.5. The crushing strength varied from 174.4 to 135.5 N when the temperature decreased from -10 to -130^{o}C. The crushing strength of ginger at room temperature was found to be 175.3N. It was observed that when the temperature lowers the crushing strength of the ginger decreases. A similar trend was reported by Mathew (1998) with black pepper subjected to crushing strength tests at different temperatures. The transition of different materials from ductile nature to brittle nature when they are cooled down to cryogenic temperature is also reported by Duthil (2011). The different test temperatures were achieved by using liquid nitrogen. Fig. 4.1 shows the plot of crushing strength versus temperature. A sudden decline in crushing strength was observed after the temperature of -40°C. This may be due to the fact that at temperatures lower than -40°C the oil particles in the raw material became solidified making the material brittle. The statistical analysis shown that the temperature have a significant effect on the crushing strength of ginger ($p<0.05$). The analysis of variance (ANOVA) to examine the effect of temperature on crushing strength and the values recorded are given in Appendix A. The relationship between the temperature and crushing strength can be expressed as;

$$\text{Crushing strength} = 0.199T + 176.57 \quad (r^2 = 0.887) \qquad \text{…..(4.1)}$$

Where, T = Temperature of the material in °C

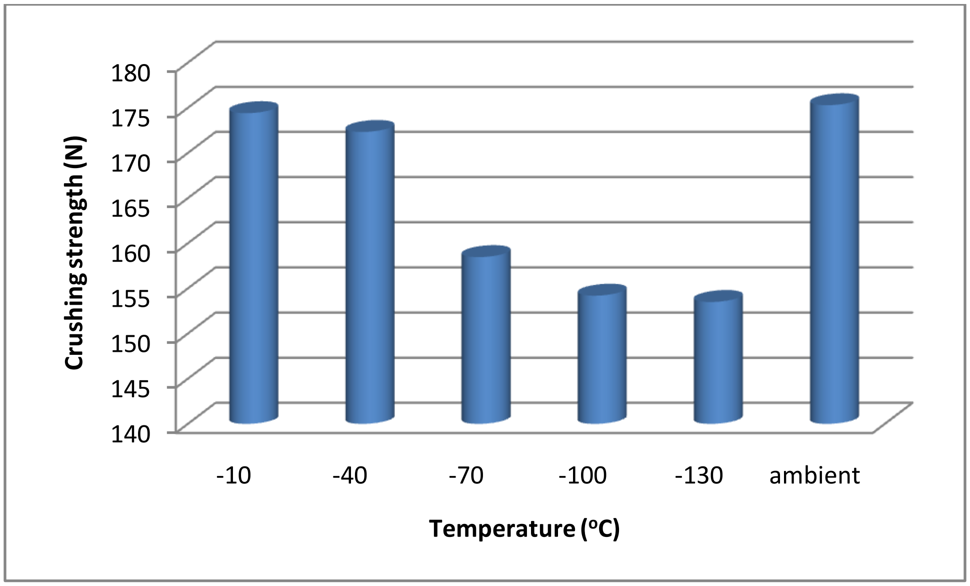


Fig. 4.1 Crushing strength of ginger at different temperatures

4.2 Studies on existing grinding methods on the quality of dried ginger powder

The term ambient grinding refers to a grinding operation which performs at room temperature and uses no temperature control or cooling methods. It starts at normal room temperature and there is a chance of temperature rise during the operation. The traditional or commercial mills do not use any kind of temperature controls or cooling methods. So it is a perfect logical assumption that performing an ambient grinding to study and have data on the effect of temperature on product quality and grinding characteristics before studying the effect of cryogenic grinding.

In this study the commonly used hammer mill was selected and the experiments were carried out. The ambient grinding of dried ginger was performed in a hammer mill explained in section 3.3. No temperature control measures were adopted during operation and the powder obtained is packed in polyethylene covers and kept under refrigeration and opened only at the time of analysis. The operations were done in triplicate and the temperature of grinding and time of grinding were recorded. The results are presented and discussed in the following section with an intention to understand the phenomena when the ginger is commercially ground.

4.2.1 Temperature rise during grinding

The temperature rise during grinding was monitored at every 30 seconds interval for the entire grinding time. The grinding was started at room temperature (29^{o}C) and the highest temperature observed was 53^{o}C and the feed temperature was 29^{o}C (room temperature). Initially the grinding temperature was the room temperature and was found to be increasing with time. The temperature at the grinding zone and the product temperature were taken separately and showed a slight difference between both. The grinding zone temperature will always be higher due to the heating up of grinder elements. The product temperature was slightly lesser than the grinding temperature because of heat transfer due to the exposure of the powder to the ambient air while exiting the grinder. Such a difference has been noticed by Murthy (2000). Pruthi and Misra (1962) reported that the temperature can go higher as 95^{o}C as the grinder elements getting heated up due to prolonged working. The Fig. 4.2 shows the temperature against time for both grinding zone and product.

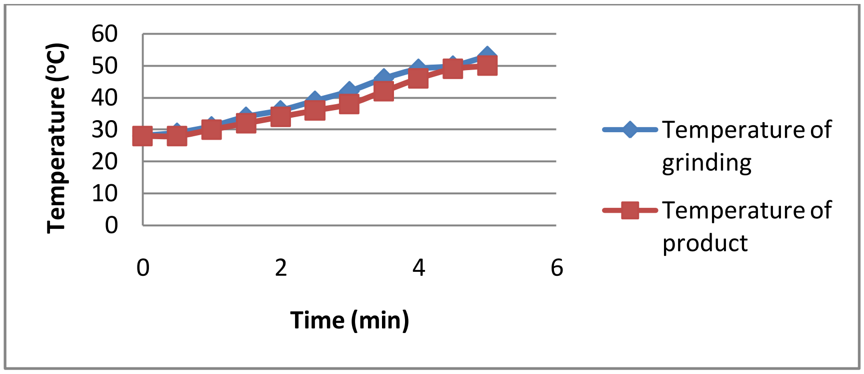


Fig. 4.2 Temperature rise during grinding

4.2.2 Grinding characteristics

The different grinding characteristics including specific energy, time of grinding and the particle size of the powder has been determined according to the methods explained in the chapter 3. The results obtained for different parameters were tabulated in Table 4.1.

Table 4.1 Different grinding characteristics of ambient grinding

Parameters	Value
Specific energy	262.4 kJ/kg
Time of grinding	157 sec
Particle size of powder	0.347 mm

4.2.3 Characteristics of ginger powder ground using existing grinding method

The various quality parameters of ginger powder ground at normal conditions have been estimated in terms of moisture content, volatile oil content, volatile oil components, oleoresin content and colour values. The procedure followed which are explained in the chapter 3. The evaluation of quality of the ambient ground powder was done by comparing it with the original sample and appended in Table 4.2. The results of various quality parameters of ambient as well as control sample were tabulated in Table 4.2.

Table 4.2 Various quality parameters of ambient ground and control samples

Quality parameter		Control sample	Ambient ground sample
Moisture content (% w.b.)		10.6	9.21
Volatile oil yield (ml/100g)		3.2	2.34
Oleoresin content (%)		7.89	7.02
Colour values	L*	65.24	61.84
	a*	5.24	6.58
	b*	30.14	26.54

The moisture content of the ambient ground powder was found to be reduced by 13.11 % of the raw material. It happened due to the evaporation of moisture content during grinding i.e., as the result of temperature rise from room temperature.

The volatile oil content in the ambient ground powder registered a reduction of 26.87% when compared with the control sample. The control sample yielded 3.2 ml/100 g of essential oil while the ambient ground sample yielded only 2.34 ml/100 g. This can be explained as the result of temperature rise due to the energy dissipation during the grinding process. It causes the volatile oil to evaporate to the surrounding atmosphere. This evaporation leads to discomfort of the laborers, create problems to eyes and even allergic in extreme cases (Murthy and Bhattacharya, 2008).

An observable change has been found in the amount of oleoresin content in the ginger powder ground at ambient conditions. The loss constitutes 11.02 % from the control sample or from the original (Sharma *et al.*, 2014). This can be attributed that the oil and fat contents in the oleoresin melts at the elevated temperatures inside the grinder and stick to its walls causing losses and even sieve clogging (Singh and Goswami, 1999).

The change in the colour values has shown that the ambient ground powder was inferior in appearance. The reduction in L* value (indicate the lightness) from the control sample shows that ambient ground product is less light in colour, the increase in a* value shows it turned more reddish and the decrease from b* value indicates that the blueness is increased. Overall, the powder turned darker.

4.3 Cryogenic grinding of dried ginger

The dried ginger was ground cryogenically using hammer mill with liquid nitrogen as cryogen. The modifications to convert the grinder to a cryogenic grinder were done as described in 3.4. The grinding has been conducted for 5 pre determined temperatures at three different feed rates as per the experimental design. The grinding characteristics such as particle size, specific energy, and time of grinding and the product characteristics like, volatile oil yield, volatile oil components, oleoresin, moisture content and colour were analyzed. It helps us to understand the effect of feed rate and grinding temperature on these parameters, and then to optimize the process for a good quality ginger powder.

4.3.1 Effect of feed rate and grinding temperature on various parameters

The various properties of grinding as well as quality regarding the grinding of ginger were investigated in detail and the observations are discussed below. The values recorded for each parameter were given in Appendix C and D.

4.3.1.1 Effect of feed rate and grinding temperature on particle size

The particle size of the ground product was determined according to the method mentioned in 3.3.1. The maximum and minimum values observed were 0.312 and 0.24 mm respectively. The highest value obtained with the highest feed rate and highest temperature. The lowest particle size was noticed for the lower feed rate and lowest grinding temperature. The particle size indicates the fineness of a powder and smaller the value finer is the powder. The fine particle size of ground spice helps in easy release of its flavor and aroma. The particle size was found to be reducing as the temperature as well as feed rate reduces from -10 to -130^{o}C and from 35 to 8 kg/h respectively. A reduction of particle size of ground product with reduction in grinding temperature was reported by Singh and Goswami (1999) from their experiments with cumin seeds. It has already been reported that the cryogenic ground product has lower particle size than non cryogenic ground products by Gopalakrishnan *et al.* (1991), and Barnwal *et al.* (2014a).

The achievement of smaller particle size might be due to the fact that at very low temperatures the moisture content and oil contents becomes crystallized in the raw material and it becomes brittle, so it is easy to break into more finer particles. The feed rate has affected the particle size in the same way as grinding temperature. The reduction in particle size with reduction in feed rate has observed by Murthy (2000) when pepper ground cryogenically. At lower feed rates the product will reside more time in the grinder so that it crumble more and resulted in the lowest particle size. The statistical analysis shows the feed rate ($p<0.0001$) and temperature ($p<0.01$) has significant effect on the particle size of dried ginger powder and implies the feed rate has a more prominent effect on particle size than grinding temperature. The change of particle size with temperature and feed rates is shown in Fig. 4.3.

4.3.1.2 Effect of feed rate and grinding temperature on specific energy

The specific energy consumption of grinding during cryogenic grinding was determined for each treatment to assess the energy requirement. The highest energy consumption was observed when the ginger ground at highest temperature and lowest feed rate and was found to be 328.4 kJ/kg. Likewise the lowest value (204.9 kJ/kg) was found for grinding operation at highest feed rate (35 kg/h) and lowest

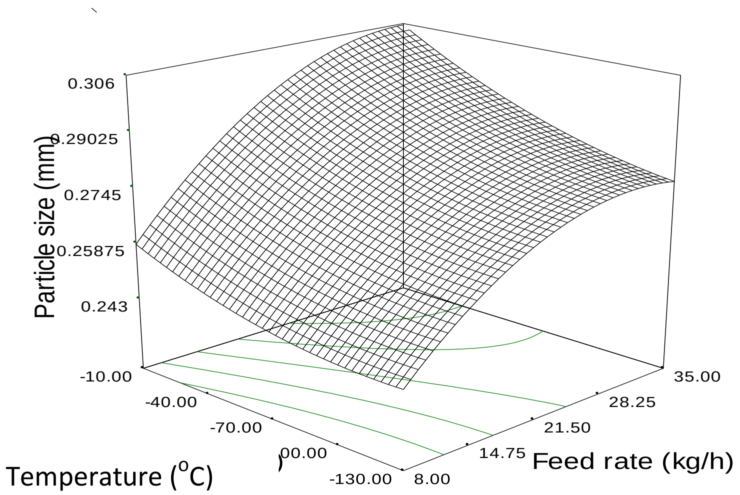


Fig. 4.3 Effect of temperature and feed rate on particle size

temperature (-130°C). The specific energy was observed to be reducing with increase in feed rate and decrease in grinding temperature. The decreasing trend of specific energy consumption with increasing feed rate can be explained as the result of fast grinding. When the grinding is faster only less time the grinder has to operate and thus less energy is required.

The linear relationship between specific energy consumption and grinding temperature is obviously due to the fact that the degree of brilttleness increases with decrease in grinding temperature i.e., the reduction in energy usage during cryogenic grinding was due to the embrittlement of raw material at lower temperatures. A brittle material can disintegrate easily with less energy than a ductile material.

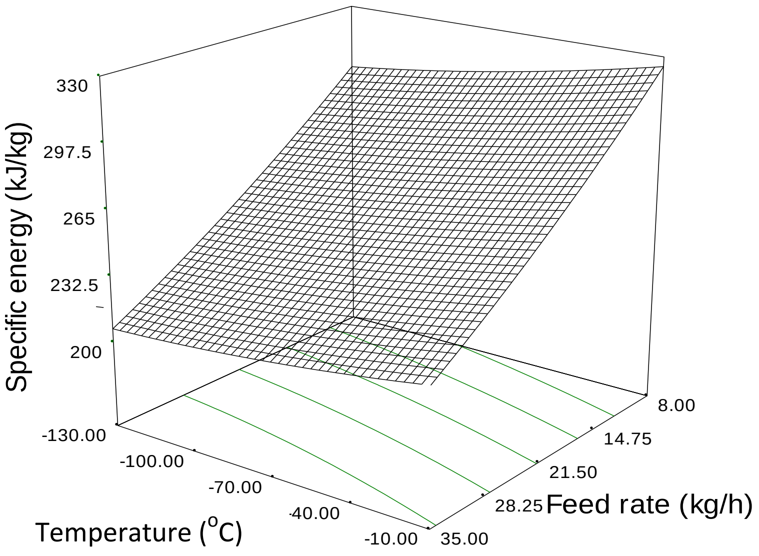


Fig. 4.4 Effect of temperature and feed rate on specific energy consumption

Similar characteristics i.e., the reduction in specific energy with the lowering of grinding temperature has been already reported by Singh and Goswami (1999), Mathew and Sreenarayanan (2007) and Barnwal *et al.,* (2014a). Lowering of specific energy with increase in feed rate was reported by Balasubhramanian *et al*. (2013). The results were statistically analysed to check the effect of grinding temperature and feed rate on the specific energy consumption and it is shown that both are significant at 1% level. Fig 4.4 shows the variation of specific energy consumption with different feed rates and temperatures.

4.3.1.3 Effect of feed rate and grinding temperature on moisture content

Moisture content is a critical factor which affects the products shelf life. The moisture content of the ground product was determined using co-distillation with toluene as per the method described in 3.2.1 using Dean-Stark apparatus. The highest value of moisture content was 10.58 and the lowest was 10.2% (w.b.). The results show that the moisture content was increasing with, decreasing grinding temperature and there was not changing with feed rate. During cryogenic grinding, the cold and inert atmosphere created by liquid nitrogen prevents the evaporation of moisture from the product. Thus the end product retains almost all the moisture content as the raw material.

The analysis of variance indicated that temperature ($p<0.001$) has significant effect on the moisture content of the material. A change in moisture content with different cryogenic treatments was shown in Fig. 4.5.

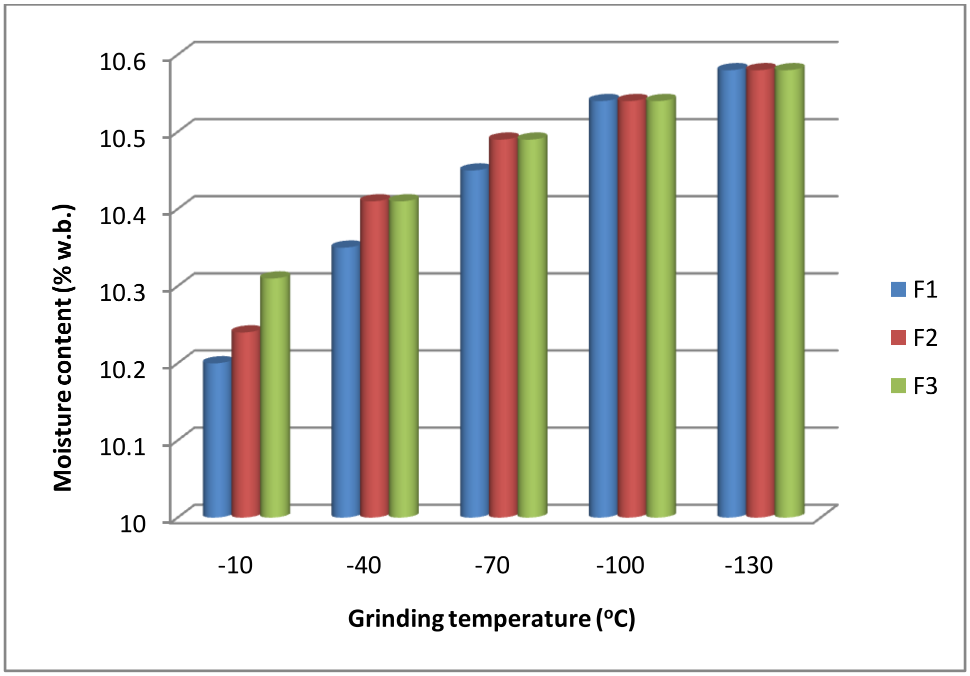


Fig. 4.5 Change in moisture content with grinding temperature and feed rate

4.3.1.4 Effect of feed rate and grinding temperature on volatile oil yield

The volatile oil content of a spice powder indirectly measures its market value. Higher the volatile oil content higher its flavor and aroma and in turn higher its quality. The volatile oil content of the powder is determined by hydro distillation method as explained in 3.3.2. The highest and lowest yield of oil observed was 3.16 and 2.9 ml/100 g and respectively. The cryogenic grinding of ginger at three different feed rates and five different temperatures shown that the yield of essential oil was increasing, with decreasing grinding temperature and not changing considerably with increase in feed rate. The highest yield was observed in the F3T5 sample i.e. when the ginger ground at -130^{o}C and 35 kg/h. Likewise the lowest yield was obtained in the treatment F1T1 i.e. when ground at lowest temperature and high feed rate. It was found that grinding temperature has a linear relationship with essential oil yield.

The essential oil in the spices is volatile in nature as they contain low boiling components. They will vaporize when they exposed to temperature. The reason for the increase in volatile oil yield with decrease grinding temperature might be explained as during cryogenic grinding, the oil containing cell in the raw material (dried ginger) gets crushed under mechanical force and the oil bodies will be freed. But the extreme cold and inert atmosphere in the grinding zone prevents the vaporization of volatile oil to the surrounding atmosphere results in retention in the end product. There was a slight increase in the oil yield observed after a grinding temperature of -40^{o}C. This increase may be due to at temperatures lower than -46^{o}C the oil particles become solidified in the spice so that it crumbles easily and reducing the oil loss further i.e. yielding more oil. The essential oil yields for different treatments were shown in Fig. 4.6.

Singh and Goswami, (1999) reported similar characteristics with the cryogenic grinding of cumin seeds. They observed an increase in cumin volatile oil content with decrease in cryogenic grinding temperatures (from -70 to -160^{o}C). This trend has also been observed by various researchers and reported that the grinding of spice at cryogenic conditions helps in retaining the volatile oil and reduces its loss to a great extent (Gopalakrishnan *et al*., 1991, Saxena and Soni, 2013, and Sharma *et al.,* 2014).

The yield of volatile oil from different treatments were analysed statistically to understand the effect of feed rate and grinding temperature on volatile oil yield. The analysis shown that temperature ($p<0.001$) has significant effect on the volatile oil yield. The Fig. 4.6 shows the yield of volatile oil for different cryoground samples. The ANOVA table to examine the effect of both parameters on volatile oil has given in appendix.

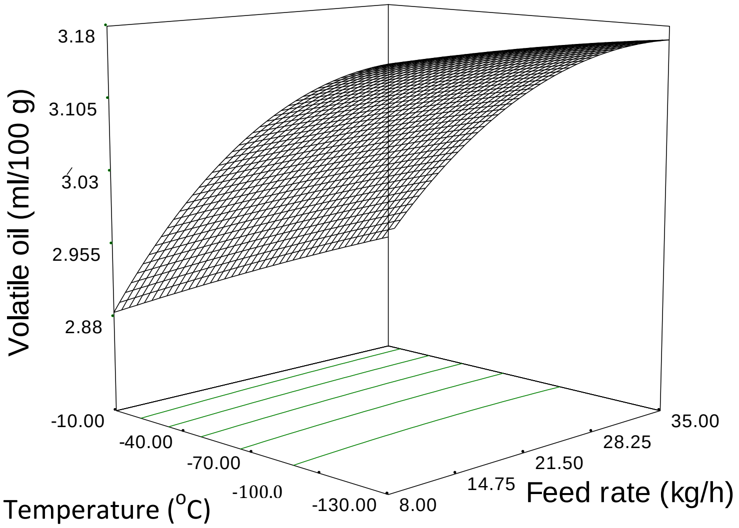


Fig. 4.6 Yield of volatile oil for different cryoground samples

4.3.1.5 Gas chromatographic (GC) analysis of ginger essential oil

The GC analysis of ginger essential oil obtained from different cryogenic treatments has been conducted using Shimadzu GC-17A. The test procedure and test conditions were followed as described in 3.3.3. The components analyzed were zingiberene, citral a (geranial) and citral b (neral). The highest retention values of zingiberene, geranial and neral were found to be 31.2, 4.67 and 2.25% and the lowest values obtained were 30.4, 4.02 and 1.89% respectively. The citrals shows an increasing trend for decreasing grinding temperature. But zingiberene found to show no particular trends with changes in feed rate and grinding temperature. The percentage retention of each constituent in ginger oil from various treatments was given in Table 4.3. The zingiberene could be identified from the chromatogram easily as it is being the highest peak. The peaks for citrals were between 11.4 to12.8 min.

Zingiberene was analyzed since it is being the major sesquiterpene hydrocarbon constituent present in the ginger essential oil. The dominance of sesquiterpenes among the volatile oil constituents particularly zingiberene, is considered as a characteristic of ginger (Zachariah, 2008). The higher retention of zingiberene indicates that the volatile oil is rich in its characteristic odour. The retention of sesquiterpenes in black pepper affected by feed rate and grinding temperature was reported by Murthy and Bhattacharya (2000). The analysis of variance to evaluate the effect of grinding temperature and feed rate on retention of zingiberene shows that both the parameters does have a significant on it.

The citral a (geranial) and citral b (neral) are the oxygenated monoterpenes present in the volatile oil of ginger providing a citrus-like odour. And these low-boiling monoterpanes hydrocarbons and oxygenated compounds attributed to the characteristic flavor of ginger essential oil. The amounts of citrals are changing with grinding parameters but not at a noticeable level. The statistical analysis shows that the grinding temperature and feed rate does not have significant effect on the retention of citral a and citral b.

4.3.1.6 Effect of feed rate and grinding temperature on time of grinding

The time of grinding has been recorded using a stop watch for each grinding operation and the effect of grinding parameters on grinding time was analysed. The

Table 4.3 Percentage retention of zingiberene, citral a and citral b

Grinding parameters		Constituents		
Feed rate (kg/h)	Grinding temperature (^oC)	Zingiberene	citral a (Geranial)	citral b (Neral)
8	-10±5	30.65	4.7	2.21
	-40±5	30.72	4.32	2.03
	-70±5	30.96	4.56	1.98
	-100±5	31.2	4.21	2.07
	-130±5	31.1	4.57	2.1
20	-10±5	30.6	4.62	1.99
	-40±5	30.69	4.52	1.89
	-70±5	30.84	4.63	2.14
	-100±5	31.2	4.53	2.17
	-130±5	31.2	4.54	2.14
35	-10±5	30.4	4.42	2.13
	-40±5	30.74	4.27	2.18
	-70±5	30.78	4.67	2.2
	-100±5	30.59	4.49	2.24
	-130±5	31.0	4.02	2.25
S.D		0.84	0.17	0.90
CV		0.27	3.88	4.24

longest and the shortest time was found to be 498 and 458 seconds for a feed rate of 8 kg/h and 201 and 187 seconds for a feed rate of 20 kg/h and 135 and 115 seconds for a feed rate of 35 kg/h. The time of grinding was observed to be reducing with decreasing grinding temperature and increasing feed rate.

When the raw material cooled down to subzero temperatures it becomes brittle. The transition of a material from ductile nature to brittle nature enables it to grind easily with less energy and to more consistent size (Sharma *et al.*, 2014). This might be the reason for the reduction of grinding time when the grinding temperature reduces. It is obvious that the grinding time is indirectly proportional with the feed rate i.e. as the feed rate decreases grinding time increases (significance $p<0.0001$for feed rate and p<0.001 for grinding temperature). The Fig. 4.7 shows the time required for grinding 1 kg of feed at different grinding conditions.

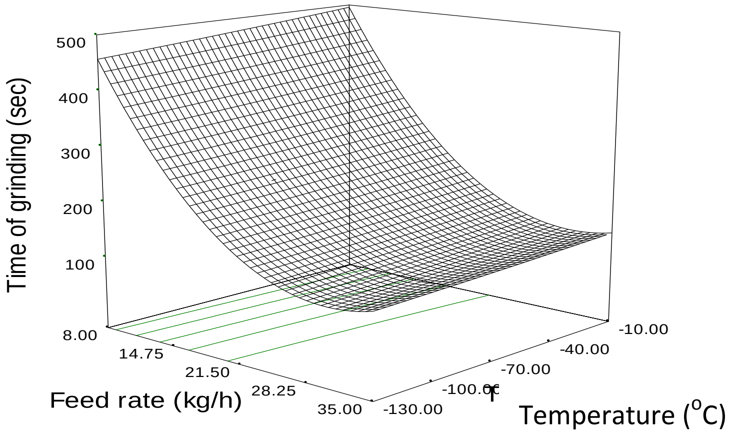


Fig 4.7 Time required for grinding 1 kg of feed at different grinding conditions

4.3.1.7 Effect of feed rate and grinding temperature on recovery of oleoresin

The ginger oleoresin contains the pungent principles of the spice and hence it is an important quality attribute. The oleoresin content in the dried ginger powder was estimated using solvent extraction (method cited in 3.3.4). The highest and lowest value for oleoresin observed were 7.76 and 7.12% for a feed rate of 8 kg/h, 7.72 and 7.08 % for a feed rate of 20 kg/h, and 7.79 and7.14 % for a feed rate of 35 kg/h. The highest yield of oleoresin was obtained with a feed rate of 35 kg/h and a grinding temperature of -130 ± 5^{o}C. And the lowest value was achieved from a combination of federate 20kg/h and a grinding temperature of -10^{o}C. The yield of oleoresin was tend to be increasing with decrease in grinding temperature and shows no particular trend with change in feed rate.

The ginger oleoresin is a dark golden-brown viscous oil and constitutes of volatile oil (20-25%) and pungency stimulating compounds (25-30%), gingerols with minor amount of shagols (Govindarajan, 1982). The grinding temperature was found to be affecting the yield of oleoresin and have a linear relationship. The increased retention of oleoresin in cryo ground samples were previously reported by Sharma *et al*. (2014). The Fig. 4.8 shows the oleoresin retention in various cryogenic ground samples. The data were analysed statistically and shown that only temperature have effect on the yield of oleoresin ($p<0.001$).

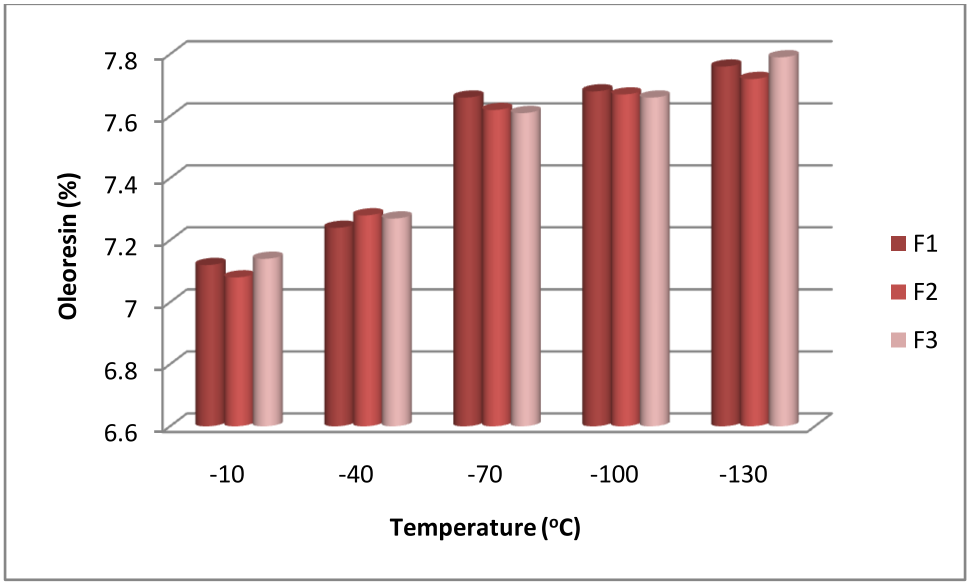


Fig. 4.8 Oleoresin recovery from different treatments

4.3.1.8 Effect of feed rate and grinding temperature on colour of ginger powder

Colour of the spice powder is one of the chief factors which draw the consumer attention first. The eye appealing colour of spice powder fetches good price to the manufacturer. The lowest and highest L* value was found to be 69.99 and 70.24 for a feed rate of 8 kg/h, 69.8 and 69.98 for a feed rate of 20 kg/h, 69.36 and 70.23 for a feed rate of 35 kg/h. The L* value indicate the luminance. It varies from darkness to lightness (0 to 100 and 100 indicates white). The lightness was found to be increasing with lowering the grinding temperature but observed no specific change with feed rate. The best L* values were found when the ginger ground at the lowest grinding temperature. The analysis of variance shows the grinding temperature has significant effect on L* value ($p<0.001$).

The highest and lowest a* value was found to be 4.03 and 3.79 for a feed rate of 8 kg/h, 3.92 and 3.75 for a feed rate of 20 kg/h, and 3.89 and 3.64 for a feed rate of 35 kg/h. The statistical analysis shows that both temperature and feed rate have no significant effect on a* value. The b* value indicates the yellowness of the powder. The highest value was found to be 31.25 for a feed rate of 35kg/h and a grinding temperature of -130^{o}C. The lowest value observed was 29.77 for a feed rate 8 kg/h and a grinding temperature -100±5^{o}C. The b* value also found to have not changing with feed rate or grinding temperature. The Table 4.4 shows the colour values of cryoground ginger powder. The Fig 4.9 shows the colour of powder ground at different temperatures.

Table 4.4 Colour values of cryoground ginger powder

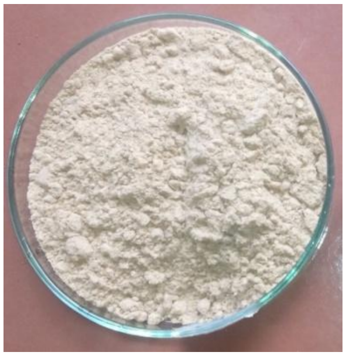
Feed rate (kg/h)	Grinding temperature (^{o}C)	Colour values		
		L*	a*	b*
8	-10±5	68.66	4.01	30.61
	-40±5	68.8	4.03	30.83
	-70±5	68.99	3.94	30.32
	-100±5	69.99	3.79	29.77
	-130±5	70.24	3.89	30.54
20	-10±5	69.24	3.92	30.62
	-40±5	69.65	3.87	30.72
	-70±5	68.45	3.81	30.41
	-100±5	69.8	3.89	30.21
	-130±5	69.99	3.75	30.85
35	-10±5	69.16	3.89	30.5
	-40±5	69.21	3.87	30.6
	-70±5	69.29	3.8	30.72
	-100±5	69.36	3.88	30.95
	-130±5	70.23	3.64	31.25
S.D		0.38	0.069	0.30
C.V		0.54	1.80	0.97

Ground at -10°C

Ground at -40°C

Ground at -70°C

Ground at -100°C



Ground at -130°C

Fig. 4.9 Colour of cryoground ginger powder

4.3.1.9 Functional properties cryoground powders

The functional properties of cryoground powders have been analysed as per the procedures described in chapter 3. The properties checked were water activity, bulk density and flowabllity. The results obtained were tabulated in Table 4.5. The highest values for water activity, bulk density and flowabllity were 0.762, 357.1 kg/m^3 and 54.2 and the lowest values observed were 0.646, 330.6 kg/m^3 and 47.9 respectively.

The water activity was found to be increased as the grinding temperature reduces and was increasing with increase in feed rate. It may be due to the fact that the retention of moisture content was increasing at lower temperatures. The statistical analysis proved that the temperature of grinding has significant effect ($p<0.001$) on water activity. The same effect of grinding temperature on water activity has reported by Mallappa *et al.* (2015).

The bulk density was found to be decreasing with decrease in grinding temperature. This can be explained in terms of moisture content. The moisture content and grinding temperature has linear relationship. The decreasing trend in bulk density indicates that the increase in volumetric expansion in the sample is higher than weight with the increase in moisture content. A similar decreasing trend for bulk density was reported by Barnwal *et al.* (2014b). The analysis of variance has shown that the effect of temperature on bulk density was significant ($p<0.01$).

The flowability of the ginger powder was analysed to understand its flow properties. The flowabilty was determined in terms of angle of repose. The value ranged from 47.9 to 54.2^{o}. A powder with this range of flowability falls under passable or poor category. The different functional properties of cryoground powder were given in Table 4.5.

Table 4.5 Functional properties of cryoground powder

Feed rate (kg/h)	Grinding temperature (^{o}C)	Functional properties		
		Water activity (a_w)	Bulk density kg/m^3	Flowabllity (degree)
8	-10±5	0.646	357.1	49.6
	-40±5	0.654	357.1	50.3
	-70±5	0.703	344.8	48.1
	-100±5	0.709	344.8	54.2
	-130±5	0.749	333.3	51.3
20	-10±5	0.681	351.4	48.2
	-40±5	0.691	347.4	51.3
	-70±5	0.712	337.9	49.4
	-100±5	0.732	334.8	53.1
	-130±5	0.753	334.5	53.4
35	-10±5	0.72	348.3	47.9
	-40±5	0.724	348.3	52.3
	-70±5	0.728	333.3	50.8
	-100±5	0.75	332.1	50.4
	-130±5	0.762	330.6	52.1

4.3.2 Optimization of cryogenic process parameters

The optimization of the process parameters of cryogenic grinding process was done with the help of statistical software Design Exert (version 7.0.0). The procedure adopted was as explained in chapter 3. The numerical optimization using software given one best solution with a desirability 0.797. The combination of feed rate and grinding temperature of the optimized process were 35kg/h and -130^{o}C, respectively.

4.3.3 Comparison of ambient ground and cryogenic ground powders

The powder obtained from optimized cryogenic grinding process was taken to compare with the powder obtained from ambient grinding operation. The ambient grinding was performed in the same hammer mill with the optimized feed rate.

4.3.2.1 Particle size

The particle size of powder obtained from ambient grinding and cryogenic grinding were observed as 0.347 and 0.24 mm respectively. This change may be explained as the result of embrittlement of raw material at grinding zone when it is performed cryogenically. The extremely low temperature in the grinder turns the ductile material into brittle so that it crumbles easily permitting to a finer and more consistent particle size. Barnwal *et al.* (2014a) compared the particle sizes of both ambient ground and cryoground cinnamon and turmeric samples and observed the same trend.

4.3.2.2 Specific energy

The specific energy of ambient grinding and cryogenic grinding for dried ginger were obtained as 262.7 and 210.3 kJ/kg. Cryogenic grinding uses only 80% of the energy consumed by the ambient grinding operation. It implies that the cryogenic grinding requires less specific energy than grinding at ambient conditions. The less specific energy consumption by cryogenic grinding when compared to ambient grinding was previously reported by Barnwal *et al.* (2014a).

4.3.2.3 Time of grinding

The time taken for grinding of a predetermined quantity of raw material was compared for ambient and cryogenic grinding with the same feed rate. The time of grinding was 157 and 122 seconds for ambient and cryogenic grinding, respectively. The saving in grinding time is in tune of 23%. This may be occurring due to the solidification of oil particles along with the solidification of moisture content in the raw material increases the degree of brittleness and enable the grinding process smoother (Singh and Goswami, 1999). At lower temperatures, the solidification of fat contents occurs and it reduces the sieve clogging. All these factors account for a smoother grinding and results in a less time of grinding.

4.3.2.4 Volatile oil

The volatile oil yield of control (original), ambient and cryogenic ground samples were 3.2, 2.34 and 3.16 ml/100 g respectively. The loss of volatile oil of ginger when it is ground at ambient conditions is about 26.8% of the oil contained in the raw material. The reduction in the oil yield is obviously due to the heat generation in the grinding zone during ambient grinding. But the loss of volatile oil content when it ground cryogenically was only 1.25% i.e. the cryogenic ground sample retains 98.7% of the volatile oil of raw material in the powder. The increase in volatile oil content in cryoground over non cryoground samples was 25.6%. Similar results were obtained when the oil yield form cryogenic ground samples compared with ambient ground samples by Saxena and Soni (2013) and Sharma *et al.* (2014) for coriander and cumin seeds.

4.3.2.5 Volatile oil components

The GC analysis was carried out for essential oil from control sample, ambient and cryogenic ground samples. The major contents like zingiberene, geranial and neral were assessed. Table 4.6 shows the retention of different constituents in various samples.

Table 4.6 Volatile oil components from different ginger samples

Essential oil from	Zingiberene	Citral a (Geranial)	Citral b (Neral)
Raw ginger	30.61	4.6	2.26
Ambient ground ginger	30.53	3.84	1.87
Cryo ground ginger	30.57	4.59	2.24
S.D	0.12	0.53	0.26

Zingiberene, the major constituent found in abundant is a high boiling sesquiterpene hydrocarbon. It is found to have not much affected by the temperature. But the oxygenated monoterpenes like citrals a and b (geranial and neral respectively) found to be affected with grinding temperature as they are being low boiling components. The decrease in geranial content was 16.5% from the original sample when it is ground at ambient conditions. But this decrease is negligible in the case of cryoground samples. The neral content was found to be reduced by 17.2% from the original but in cryoground samples it was observed as 99% of neral was retained.

It infers that the grinding of ginger at ambient conditions reduces the important volatile oil constituents which lead to reduction in the characteristic aroma of ginger oil. It might be owing to the fact that the higher temperature in the grinder leads to the vaporization of low boiling constituents in ginger oil. The essential oil from ambient ground samples were found to contain less citrals, and can infer that it is inferior in quality. In cryoground samples, the extreme low temperature in the grinding zone prevents the evaporation of low boiling constituents and may leads to their higher retention. The cryogenic grinding lead to higher retention of volatile oil constituents was previously reported by Murthy (1999) and Mathew (1998).

The gas chromatogram for oil obtained from raw material, ambient and cryo ground samples were given in Fig.4.10, Fig.4.11 and Fig.4.12 respectively. The major compound zingiberene can be identified easily as it has the highest peak.

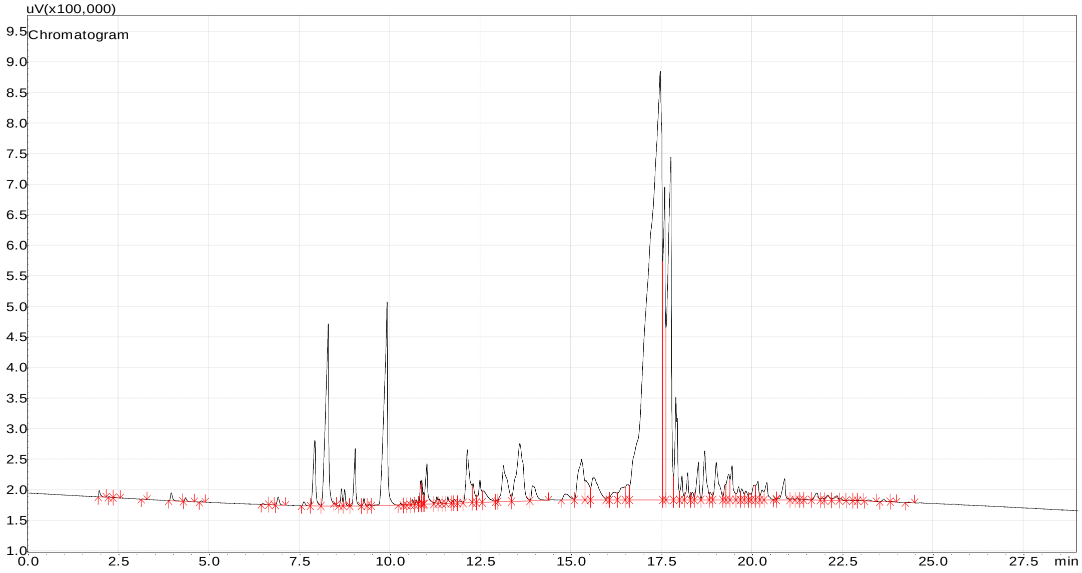


Fig. 4.10 Gas chromatogram of essential oil from original sample

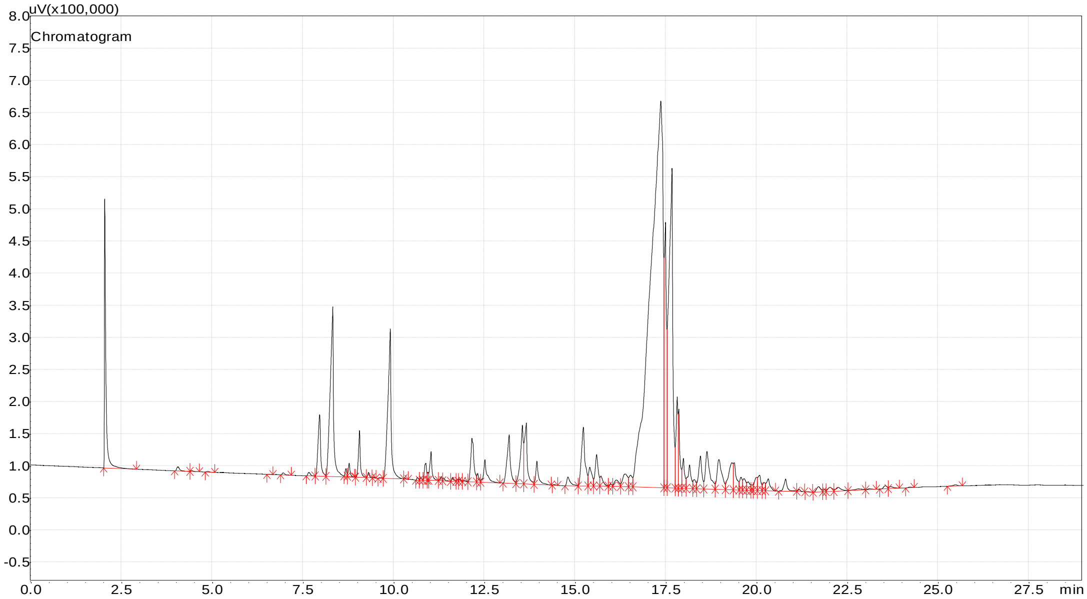


Fig. 4.11 Gas chromatogram of essential oil from ambient ground sample

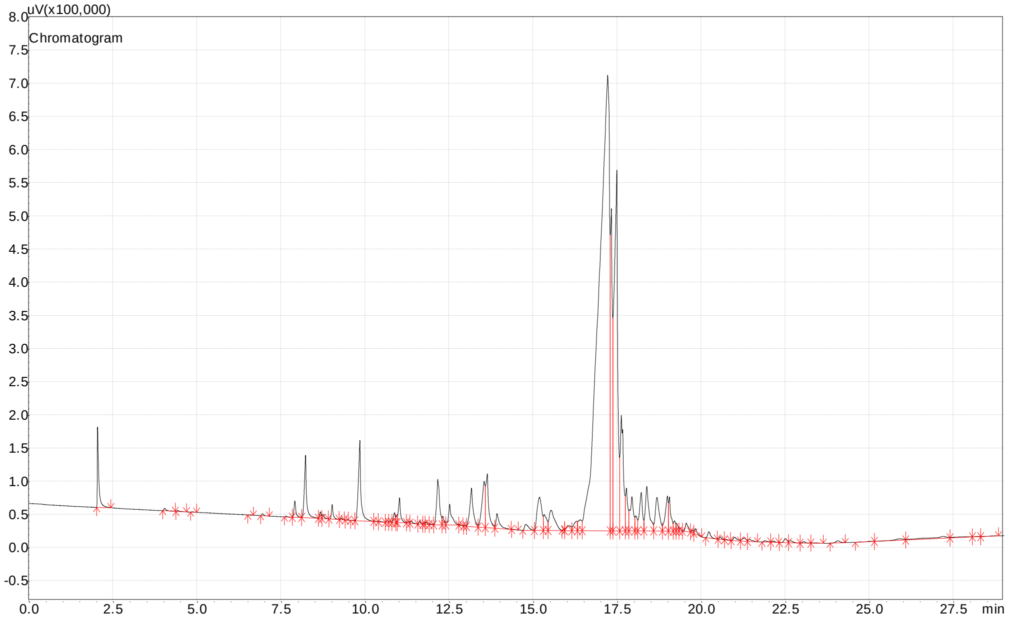


Fig. 4.12 Gas chromatogram for essential oil from optimized cryoground sample

4.3.2.6 Oleoresin

The yield of oleoresin from original, ambient and cryogenic ground samples and the percentage recovery were determined and tabulated in Table 4.7.

Table 4.7 Oleoresin recovery

	Oleoresin (%)	Per cent loss from original sample	Per cent retention in cryo ground over ambient ground
Original	7.89		
Ambient ground	7.02	11.02	
Cryoground	7.79	1.26	9.88

From the table it is obvious that the oleoresin content is more in cryoground samples. There is a noticeable loss in oleoresin in ginger powder when it is ground at ambient condition but the cryoground samples retains nearly 98% of oleoresin in the raw material. Sharma *et al.* (2014) compared the oleoresin yield from ambient and cryoground samples and discovered the same results. A same tendency was observed by Saxena *et al.* (2015) with coriander seeds.

4.3.2.7 Colour

The colour values obtained for ambient and optimized cryoground samples were tabulated in Table 4.8 for comparison.

Table 4.8 Comparison of colour values of ambient and cryogenic ground ginger

Ginger powder	L*	a*	b*
Ambient ground	61.84	6.58	26.54
Cryoground	69.36	3.88	30.95

It can be seen from the table that the luminance (lightness) of ambient ground sample is less compared to cryoground sample which means it is darker than the latter. The increase in a* value shows that the ambient ground powder was tend to be more reddish than the cryoground powder. The decrease in b* value of ambient ground powder indicates that its yellowness is less when compared to cryoground powder. Overall, the colour of ginger powder obtained from grinding at ambient conditions resulted in an inferior colour when compared to grinding at cryogenic conditions.

4.3.2.8 Moisture content

The moisture content of the ambient and cryogenic ground ginger powder was 9.21 and 10.58 % (w.b.). Whilst the moisture content of the raw material was 10.6% (w.b.). This drastic change in the moisture content of the ambient ground ginger powder might be owing to the fact that the heat generation in the grinder. The ambient grinding causes a reduction in moisture content by 13.1% in the powder from the raw material, but the reduction in moisture content by cryogenic grinding is only 0.18%

from the original. The increase in retention of moisture content in cryogenic ground ginger over ambient ground ginger was determined as 12.9%.

4.4 Packaging studies of ginger powder

The powder obtained from the optimized process has been selected for storage and package studies. The packaging materials selected were laminated aluminum foil, LDPE 400, LDPE 200, and LDPE 100 gauges covers. Storage studies have been carried out under ambient conditions. The different quality parameters (moisture content, volatile oil content, oleoresin and colour and functional properties like water activity, bulk density and flowability) were analysed at an interval of 30 days for 3 months. The results were presented in the following sections and the records of different parameters are given in Appendix E.

4.4.1 Moisture content

The moisture content was found to be varying during storage period. An increasing trend has been noticed in all the packages. The increase in moisture content during storage is due to the moisture ingression through the packaging material. The increase of moisture in the laminated aluminum package was found to be lesser among the other packaging materials. Similar results were obtained by Chetti *et al.* (2012) during the storage of chilli powder. A plot of change in moisture content during storage has given in Fig.4.13.

4.4.2 Volatile oil content

The three month storage shows that there is no change in volatile oil content in the powder kept in laminated aluminum foil. It might be due to the strong barrier property of the material. But in other packages a slight reduction was observed in oil content with increase in storage time. The change in the volatile oil content in different packages for three months has shown in the Fig.4.14.

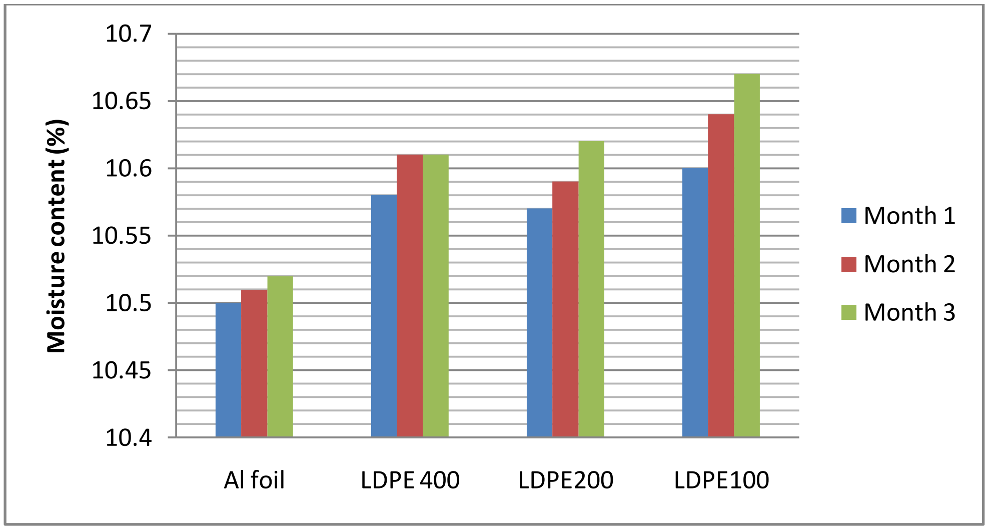


Fig. 4.13 Change in moisture content during storage

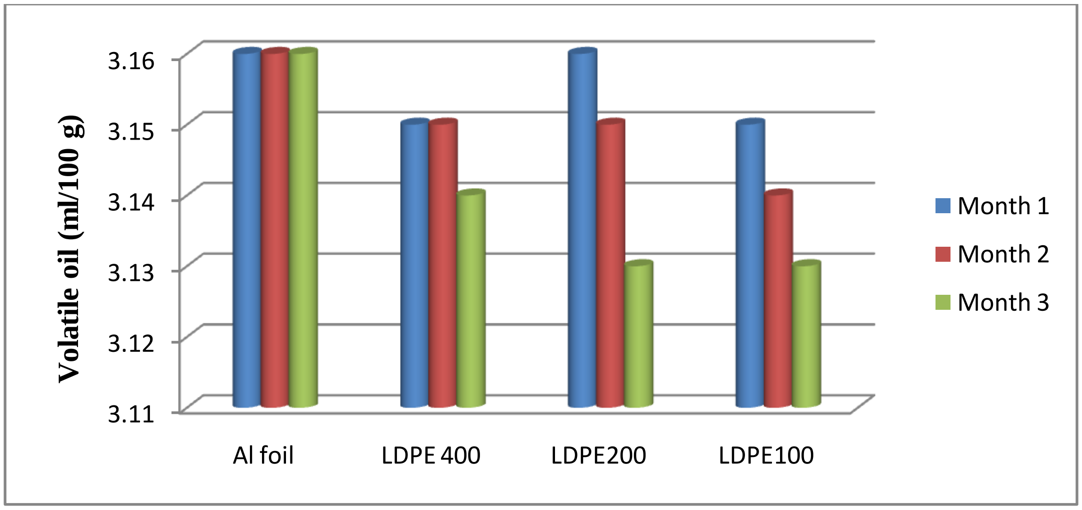


Fig. 4.14 Change in volatile oil during storage

4.4.3 Changes in the oleoresin content and colour

The three month storage and packaging study shows there was no loss of oleoresin content in the ginger powder. The loss percentage was negligible. This may be due to the reason that there was no temperature abuse to the product during storage. The result of colour values taken in an interval of one month for a duration of three months shows that the colour of the powder was not affected by the storage period or the packaging material.

4.4.4 Changes in functional properties

The results of analysis of different functional properties have given below in Table 4.9. The water activity was found to be increased throughout the storage period in all the packaging materials. The largest change was found in LDPE 100 gauge material, from 0.731 to 0.771.The increment was found negligible in ginger powder kept in laminated aluminum packages. The bulk density was found to have no significant change with the storage time in all the packaging materials. The flowability was found decreased when the angle of repose is increased from 49.4 to 50.4 degree. Negligible increase was found in powder kept in laminated aluminum package.

Table 4.9 Changes in functional properties with storage period

	Month 1	Month 2	Month 3
Water activity			
Al foil	0.715	0.723	0.72
LDPE 400	0.725	0.727	0.74
LDPE200	0.721	0.728	0.756
LDPE100	0.731	0.734	0.771
Bulk density (kg/m^3)			
Al foil	332.4	332.4	332.5
LDPE 400	333.4	332.4	332.4
LDPE200	332.2	332.2	333.4
LDPE100	333.1	333.2	333.2
Flowability			
Al foil	49.4	49.5	49.5
LDPE 400	49.8	50.1	50.1
LDPE200	50.1	50.4	50.4
LDPE100	50.2	50.3	50.4

4.4.5 Optimization of packaging material

The optimization of the best or suitable packaging material for ginger powder for a period of at least three months based on performance has been carried out using a statistical software Design expert (version 7.0.0). The optimization has done for the packaging material with minimum changes in the powder quality during storage. The packaging material was selected with ginger powder kept in it have maximum volatile oil, maximum oleoresin, minimum moisture, maximum colour minimum flowability, minimum bulk density, and minimum water activity. The best results were found for laminated aluminum foil. It infers that the packaging of ginger powder in laminated aluminum foil retained the quality attributes to the best level when compared to the other packaging materials for a period of three months.

SUMMARY AND CONCLUSIONS

CHAPTER V

SUMMARY AND CONCLUSIONS

Spices belong to important agricultural products due to its higher demand in both domestic and international markets. The Indian culinary arts will not complete without the tangy and appetizing taste of the incredible spices. The demand for spices has always been increasing as newer ways are exploring to make better use for it.

The processing of spices adds its value and enables it to export and earn foreign money. The powdering of spices helps them easy to mix with food materials and maximize the extraction of its components. Ginger is one of the major spices in India and is used widely in food as well as in traditional medicines. Ginger is widely used in the form of powder. Ginger is a highly fibrous rhizome and become too hard when dried. The process of powdering or grinding of ginger by the conventional process viz. using hammer mill, at ambient conditions takes long time and high energy causing quality loss.

Retention of biochemical quality of spices even after grinding is an important attribute of spice processing. The quality loss will not be compromised for the export as well as domestic use. Grinding is an important unit operation for spices in which higher temperature than the ambient level may affect the heat sensitive components which contribute the characteristic flavor and aroma. Keeping the importance of using ground form of ginger in ayurveda medicine and the world trade the present study was initiated to optimize the grinding parameters using cryogens.

The study includes the determination of different physical parameters of ginger, traditional and cryogenic grinding of ginger, and the packaging study of ginger powder. The result of the studies and the conclusions are mentioned here briefly.

Physical parameters like shape, hardness, moisture content, freezing point of volatile oil and crushing strength at different temperatures have been determined. Grinding of ginger with traditional method i.e. grinding using a hammer mill without any cooling or temperature controlling method was done and the grinding characteristics like particle size, specific energy and time of grinding were recorded. The powder quality like moisture content, volatile oil yield, volatile oil profile, oleoresin content and color were analysed. It was found that the temperature during grinding was raised up to 53^{o}C. The powder registered a reduction of 26.87% volatile oil and oleoresin content by11.02% from the original.

Cryogenic grinding using liquid nitrogen has been experimented to optimize the process parameters like feed rate and grinding temperature. These two parameters are found to have an effect on quality and grinding characteristics, and they are fixed as independent variables, based on literature. The data were analysed statistically to check the significance, with suitable software (Design Expert, version 7.0.0).

The change in feed rate was found to be affected the machine parameters like particle size, specific energy and time of grinding. It does not have any significant effect on major quality parameters like zingiberene content, volatile oil and colour. By considering the economy, machine parameters and quality parameters the machine running at full capacity i.e. 35 kg/h may be recommended.

The variation in test temperatures from -10 to -130^{o}C caused some variation in grinding as well as product characteristics. The temperature has a significant effect on particle size, specific energy, volatile oil, oleoresin and colour. The gas chromatographic analysis of volatile oil indicated that the zingiberene content, the high boiling sesquiterpene hydrocarbon does not vary considerably when grinding temperature reduces. Whereas the geranial and neral (low boiling monoterpenes) retained 16.9 and 16.14% respectively than the ambient ground samples.

The moisture content, water activity and flowability of ginger powder were found to be affected with the extreme low temperature of grinding. The heating effect in the conventional grinding resulted a powder with less moisture content, less water activity and good flowability.

The optimization was done with the help of statistical software Design expert 7.0.0. The method followed was general factorial. The response functions have been selected based on experimental design and the optimized condition was chosen with the maximum desirability. It was obtained that the higher feed rate (35 kg/h) is the optimum so that it gives viable machine parameters. The further decrease in grinding temperature from -130^{o}C will not result in more volatile oil yield or oleoresin because cryogenic ground powder retains it 98.7 and 99% of the raw material respectively, so that it is the optimized as the temperature for cryogenic grinding of ginger.

The ginger ground at the optimized cryogenic condition was compared with the same ground in ambient conditions. The ginger powder ground at cryogenic condition retained 26.67% more volatile oil over ambient ground powder. The same trend has been followed for oleoresin also and the percentage retention was 9.82% more than the ambient ground one. These observation shows that the cryogenic grinding reduces deterioration of ginger in terms of quality and retains the biochemical properties ginger even after grinding to a considerable level than the ginger ground with existing methods.

Finer particle size was obtained in cryogenic ground ginger (0.24 mm) than conventional grinding (0.347 mm). The reduction in specific energy consumption and time of grinding for cryogenic processing prove that it improves the grinding characteristics, make the operation smoother and viable when energy is concerned. The analysis of colour values for both the sample shows the lightness and yellowness is more for cryoground samples. The lightness of ambient ground ginger was less and redness was more inferring it is darker. Thus the cryogenic grinding of ginger is better than the conventional grinding. The experiments with different packaging materials have shown that laminated aluminum foil is suitable for long term storage of ginger powder.

From this study we can conclude that, ginger has been ground successfully by using a cryogenic grinder and the process parameters were optimized as feed rate: 35 kg/h and grinding temperature -130±5°C. Cryogenic grinding is a promising technology which results in good grinding characteristics of ginger and implementing this technology gives a powder with less quality deterioration. The following are the suggestions for future research work on the cryogenic grinding of ginger:

1. Studies may be conducted with different conditioning of ginger prior to grinding.
2. Studies may be conducted with different sieve sizes to check the effectiveness at different feed rates and moisture levels.

REFERENCES

ERENCES

[Anonymous]. 2012. Packaging of Spices. [on line]. Available: http://icpe.in/icpefoodnpackaging/pdfs/16_spices.pdf [28 Feb 2012].

[Anonymous]. 2015a. IBEF home page [on line]. Available: http://www.teacoffeespiceofindia.com/spice/spice-statistics.

[Anonymous]. 2015b. Spice Board of India [on line]. Ginger. Available: http://www.indianspices.com/spice-catalog/ginger-0[18 Nov 2015].

Adinoyi, E.H., Joseph, U.U., Chukwuma S., and. Adeyemi, O. O. 2015. Possible short term storage effect on some chemical properties of locally processed turmeric powder in South-eastern Nigeria. *Asian J. Plant Sci. and Res*. 5(7):6-12.

Al-Amin, Z.M., Thomson, M., Khaled, K., Al-Qattan, Shalaby, R.P., and Ali, M. 2006. *Br. J. Nutr*. 96: 660–666.

Ali, B.H., Blunden, G., Tanira, M.O., and Nemmar, A. 2008. Some phytochemical, pharmacological and toxicological properties of ginger (*Zingiber officinale Roscoe*): A review of recent research. *Food Chem. Toxicol*. 46(2):409-420.

Balasubramanian, S., Kumar, R., Singh, K.K., Zachariah, T.J., and Vikram. Size reduction characteristics of black pepper. 2013. *J. Spices and Aromat. Crops.* 22 (2): 138–147.

Barnwal, P., Mohite, A.M., Singh, K.K., and Kumar, P. 2014b. Selected Physico-Mechanical Characteristics of Cryogenic and Ambient Ground Turmeric. *Int. Agrophys.* 28: 111-117.

Barnwal, P., Mohite, A.M., Singh, K.K., Kumar, P., Zachariah, T.J., and Saxena, S.N. 2014a. Effect of cryogenic and ambient grinding on grinding characteristics of cinnamon and turmeric. *Int. J. Seed Spices.* 4(2):26-31.

Canovas, G.B., Rivas, E.O., Juliano, P., and Yan, H. 2005. *Food Powders: Physical Properties, Processing and Functionality*. Plenum Publishers, New York. 163p.

Chetti, M. B., deepa, G.T., Antony, T. R. Uppar, D.S. and Navalgatti, M.C. 2012. Influence of vacuum packaging and long term storage on quality of chilli powder (*Capsicum annuum L*.). J. Food Sci. and Technol. 51(10): 2827-2832.

Cleef, J.V. 1991. Powder Technology. *American Sci.* 79(4): 304-315.

Daily, J.W., Yang, M., Kim, D.S., and Park, S. 2015. Efficacy of ginger for treating Type 2 diabetes: A systematic review and meta-analysis of randomized clinical trials. *J.Ethnic Foods.* 2(1):36–43.

Duthil. P. 2011. Material Properties at Low Temperature. [on-line]. Available: https://arxiv.org/ftp/arxiv/papers/1501/1501.07100.pdf[29 Jan. 2015].

Edeskuty, F.J. and Stewart, W.F. 1996. *Safety in the Handling of Cryogenic Fluids*. Plenum Press, New York. 4p.

Geldart, D., Abdullah, E.C., Hassanpour, A., Nwoke, L.C., and Wouters. I. 2012. Characterization of powder flowability using measurement of angle of repose. *China Particuology*. 4(3): 104-107.

Ghosh, A.K., Sarkar, P., Mahmud, A. K., And Kaiyum, A.M. 2011. Gingerol might be a sword to defeat colon cancer. *Int. J. Pharma. and Bio Sci.* 2(1): 816-827.

Gopalakrishnan, M., Varma, R. L., Padmakumari, K.P., Symon, B., Umma, H., and Narayanan, C.S. 1991. Studies on cryogenic grinding of cardamom. *Indian Perfumer*. 35(1): 1-7.

Goswami, T.K. and Ghodki, B.M. 2015. Cryogenic Grinding of Cassia. In: Proceedings of International Conference on Advances in Chemical, Biological and Environmental Engineering (ACBEE). 29-30March, 2015, Singapore, pp. 242-249.

Govindarajan, V.S. 1982. Ginger – chemistry, technology and quality evaluation. *Crit. Rev. Food Sci. and Nutr.*17(1):1–96.

Guptha, S., and Ravishankar, S. 2005. A comparison of the antimicrobial activity of garlic, ginger, carrot and turmeric pastes against *E.coli* in laboratory buffer and ground beef. *Food borne Pathog Dis*. 2(4):330-340.

Habib, S.H. M., Makpol, S., Hamid, N. A. A., Das, S., Ngah, W. Z. W., and Yusof, Y. A.M. 2008. Ginger Extract (*Zingiber Officinale*) has Anti-Cancer and Anti-Inflammatory Effects on Ethionine-Induced Hepatoma Rats. *Clinics.* 63(6): 807–813.

Islam, K., Rowsni, A.A., Khan, M.M., and Kabir, M.S. 2014. Antimicrobial Activity of Ginger (Zingiber Officinale) Extracts Against Food-Borne Pathogenic Bacteria. *Int. J. Sci., Environ. and Technol.* 3(3): 867 – 871.

Jayashree, E. and Visvanathan, R. 2011a. Physical and biochemical parameters of fresh and dry ginger (*Zingiber officinale Roscoe*). *J. Spices and Aromat. Crops.* 20(1): 14-21.

Jayashree, E. and Visvanathan, R. 2011b. Mechanical and thermal properties of ginger (Zingiber officinale Rosc.) *J. Spices and Aromat. Crops*. 20(2): 60–65.

Kaleemullah, S. and Kailappan, R. 2003. Geometric and Morphometric Properties of Chillies. *Int. J. Food Prop*. 6: 481-498.

Khater, D.S. 2010. The influence of ginger as a chemopreventive agent on proliferation and apoptosis in chemically induced oral carcinogenesis. *J. Nat. and Sci.* 8(11):44-51.

Kim, N.K. and Hung, Y.C. 1994. Freeze Cracking in Food as Affected by Physical Properties. *J. Food Sci.* 59(3): 669-674.

Landwehr, D. and Pahl, M.H. 1986. Cold grinding of spices. *Int. J. Food Technol. and Food Process Eng.,* 37: 174-185.

Loncin, M. and Merson, R.L. 1979. *Food Engineering: Principles and Selected Applications*. Academic Press. New York.

Mahluji, S., Ostadrahimi, A., Mobasseri, M., Attari, V.E., and Payahoo, L. 2013. Anti-Inflammatory Effects of *Zingiber Officinale* in Type 2 Diabetic Patients. *Advanced Pharma. Bull.,* 3(2): 273-276.

Mallappa, J.M., Sharankumar, H., and Roopa, R.S. 2015. Effect of Milling Methods and its Temperature on Quality Parameters of Byadagi Chilli: With Emphasis on Cryogenic Grinding. *Res. J. Eng. Sci*. 4(3): 1-5.

Mathew , S.M. and Sreenarayanan , V.V. 2007. Study on grinding of black pepper and effect of low feed temperature on product quality. *J. Spices and Aromat. Crops*. 16(2): 82-87.

Mathew, S.M. 1998. Studies on cryogenic grinding of pepper. Ph.D thesis, Tamil Nadu Agricultural University, Coimbatore. 16p.

Meghwal, M. and Goswami, T.K. 2010. Cryogenic Grinding of Spices is a Novel Approach Whereas Ambient Grinding Needs Improvement. Continental *J. Food Sci. and Technol.* 4: 24 – 37.

Meghwal, M. and Goswami, T.K. 2011. Thermal Properties of Black Pepper and Its Volatile Oil. *Int. J. Adv. Biotechnol. and Res*. 2(3): 334-344.

Meghwal, M. and Goswami, T.K. 2014. Effect of grinding methods and packaging materials on fenugreek and black pepper powder quality and quantity under normal storage conditions. *Int. J. Agric. and Biol. Eng.* 7(4):106-113.

Murthy, C.T. 2001. Cryogenic Size Reduction and Engineering Properties of Black Pepper. Ph.D thesis, University of Mysore, Mysore, 38p.

Murthy, C.T. and Bhattacharya, S. 2008. Cryogenic grinding of black pepper. *J. Food Eng*. 85:18–28.

Nampoothiri, S.V., Venugopalan, V.V., Joy, B., Sreekumar, M.M., and Menon, A.N. 2012. Comparison of Essential oil Composition of Three Ginger Cultivars from Sub Himalayan Region. *Asian Pacific J. Tropical Biomedicine*. 1347-1350.

Nwaoha, M., Elizabeth, I., Okafor, Ifeanyi, G., and. Veronica, A.O. 2013. Production of oleoresin from ginger (Zingiber officinale) peels and evaluation of its antimicrobial and antioxidative properties. *African J. Microbiol. Res.* 7(42): 4981-4989.

Onyenekwe, P.C. and Hashimoto, S. 1999. The composition of the essential oil of dried Nigerian ginger (Zingiber officinale Roscoe). *Eur. Food Res. and Technol*. 209(6): 407–410.

Pandian, N.K.S., Dhananchezhiyan, P., and Parveen, S. 2013. Physical and Engineering Properties of Tamarind Fruit. *Int. J. Sci.Eng. and Technol.* 2(11):1083-1087.

Phoung, S., and Sertwasana, A. 2010. Spray-drying of ginger juice and physicochemical properties of ginger powders. Science Asia. 36:40-45.

Pruthi, J.S. 1980. *Spices and Condiments-Chemistry, Microbiology and Technology*. Academic Press, New York. 207.

Pruthi, J.S. 1999. *Quality Assurance in Spice and Spice Products- Modern Methods of Analysis*. Allied Publishers Limited, New Delhi, 59p.

Pruthi, J.S., and Misra, B.D. 1963. Physical, Chemical and Microbiological Changes in Curry Powders During Drying, Milling and Mixing Operation. *Spice Bull.* 3:3-5.

Pruthi, J.S. 1993. *Major Spices of India Crop Management and Post Harvest Technology*. Publications and Information Division, Indian Council for Agricultural Research, New Delhi. 274p.

Russo, J.R. 1976. Cryogenic Grinding and Carousel Material Handling. *J. Food Eng*. 3:33-35.

Sahay, K.M. and Singh, K.K. 2001. Unit Operations of Agricultural Processing, (2nd Ed). Vikas Publishing House Pvt. Ltd., New Delhi, 221p.

Sasidharan, I. and Menon, A.N. 2010. Comparative Chemical Composition and Antimicrobial Activity Fresh & Dry Ginger Oils (Zingiber Officinale Roscoe). *Int. J. Curr. Pharma. Res*. 2(4): 40-43.

Saxena, R. and Soni, A. 2013. Cryogenic Grinding: A Physical Technique To Retain Volatile Content In Natural Products. *Int. J. Mod. Phys*. 22: 589–592.

Saxena, R., Rathore, S.S., Barnwal, P., Soni, A., Sharma, L., and Saxena, S.N. 2013. Effect of cryogenic grinding on recovery of diosgenin content in fenugreek (*Trigonella foenum- graecum* L.) genotypes. *Int. J. Seed Spices.*3(1):26-30.

Saxena, S.N., Sharma, Y.K., Rathore, SS., Singh, K.K., Barnwal, P., Saxena, R., Upadhyaya, P., and Anwer, M. M. 2015. Effect of cryogenic grinding on volatile oil, oleoresin content and anti-oxidant properties of coriander (*Coriandrum sativum L.*) genotypes. *J. Food Sci. Technol.* 52(1):568–573.

Sharma, L.K., Agarwal, D., Sharma, Y., Rathore, S.S., and Saxena, S.N. 2014. Cryogenic grinding technology enhances volatile oil, oleoresin and antioxidant activity of cumin (*Cuminum cyminum L.*). *Int. J. Seed Spices.* 4(2):68-72.

Sindhu, G.K., Arora, S., and Bhatia, S. 2010. Effect of storage methods on quality of turmeric powder. *J. Res. Punjab Agricultural University*. 47(12):76-81.

Singh, K.K. and Goswami, T.K. 1999. Design of a Cryogenic Grinding System for Spices. *J. of Food Eng*. 39: 359-368.

Singh, K.K. and Goswami, T.K. 2000. Cryogenic grinding of cloves. *J. Food Processing and Preservation.*

Singh, V.B. and Singh, K.1996. *Spices*. New Age International Pvt. Ltd. Publishers, New Delhi, 61p.

Spices Board, 2015. Review on Export Performance of Spices from India 2014-15[on line]. Available: http:// http://www.indianspices.com/export/major-itemwise-export [20 Nov 2015].

Spices Board, 2016. Review of Export performance of Spices during 2015-16. [on-line]. Available: http://www.indianspices.com/export/major-itemwise-export [5 Jul 2016].

Tan, B.K.H. and Vanitha, J. (2004). Immunomodulatory and Antibacterial Effects of Some Traditional Chinese Medicinal Herbs: A Review. *Curr. Med. Chem.*, 11(11):1423-1430.

Wilczek, M., Bertling, J., and Hintemann, D. 2004. Optimised Technologies For Cryogenic Grinding. *Int. J. Miner. Process*. 74:425-434.

Zachariah, T.J. 2008. Ginger. In: Parthasarathy, V.A. and Chempakam, B. (eds), Chemistry of Spices. CAB International, United Kingdom, pp 70-75p. Available: http://catbull.com/alamut/Bibliothek/Chemistry_of_Spices.pdf [13 Aug 2011].

APPENDIX-A

Table A.1 Different temperatures measured during cryogenic grinding of ginger

Feed rate (kg/h)	Temperature (^oC)		
	Feed	Grinding zone	Product
8	29±2	-10±5	-7±5
8	29±2	-40±5	-35±5
8	29±2	-70±5	-68±5
8	30±2	-100±5	-97±5
8	30±2	-130±5	-125±5
20	29±2	-10±5	-6±5
20	29±2	-40±5	-35±5
20	30±2	-70±5	-67±5
20	29±2	-100±5	-94±5
20	29±2	-130±5	-127±5
35	30±2	-10±5	-6±5
35	29±2	-40±5	-34±5
35	30±2	-70±5	-68±5
35	29±2	-100±5	-96±5
35	29±2	-130±5	-126±5

Table A.2 Crushing strength of dried ginger at different temperatures

Test temperature (^oC)	-10	-40	-70	-100	-130
Crushing strength (N)	174.41	172.33	158.47	154.2	153.5

Table A.3 ANOVA to examine the effect of temperature on crushing strength of ginger

	df	ss	ms	F-value
Regression	1	359.40025	359.40025	23.86771
Residual	3	45.17403	15.05801	
Total	4	404.57428		

APPENDIX- B

Table B.1 ANOVA for effect of feed rate and grinding zone temperature on volatile oil

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	0.15	6	.025	344.71	<0.0001	8.66	0.28
Feed rate	2.560E-003	2	1.280E-003		0.421		
Temperature	0.15	4	0.036	17.86	<0.0001		
Residual	5.733E-004	8	716E-005	508.14			
Cor total	0.15	14				Significant	

Table B.2 ANOVA for effect of feed rate and grinding zone temperature on Particle size

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	5.175E-003	6	8.626E-004	32.11	< 0.0001	5.183E-003	1.90
Feed rate	4.248E-003	2	2.124E-003	79.06	< 0.0001		
Temperature	9.271E-004	4	2.318E-004	8.63	0.0053		
Residual	2.149E-004	8	2.687E-005				
Cor total	5.390E-003	14				Significant	

Table B.4 ANOVA for effect of feed rate and grinding zone temperature on oleoresin

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	0.95	6	0.16	226.77	< 0.0001	0.026	0.35
Feed rate	1.213E-003	2	6.067E-004	0.87	0.4556		
Temperature	0.95	4	0.24	339.71	< 0.0001		
Residual	5.587E-003	8	6.983E-004				
Cor total	0.96	14				Significant	

Table B.4 ANOVA for effect of feed rate and grinding zone temperature on Moisture content

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	0.19	6	0.032	21.43	0.0002	0.039	0.37
Feed rate	7.293E-003	2	3.647E-003	2.44	0.1492		
Temperature	0.19	4	0.046	30.92	< 0.0001		
Residual	0.012	8	1.497E-003				
Cor total	0.20	14				Significant	

Table B.5 ANOVA for effect of feed rate and grinding zone temperature on Specific energy

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	23889.81	6	3981.64	492.49	< 0.0001	2.84	1.08
Feed rate	22914.29	2	11457.14	1417.15	< 0.0001		
Temperature	975.53	4	243.88	30.17	< 0.0001		
Residual	64.68	8	8.08				
Cor total	23954.49	14				Significant	

Table B.6 ANOVA for effect of feed rate and grinding zone temperature on Time of grinding

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	3.430E+005	6	57164.49	2371.97	< 0.0001	4.91	1.84
Feed rate	3.415E+005	2	1.708E+005	7085.20	< 0.0001		
Temperature	1480.40	4	370.10	15.36	0.0008		
Residual	192.80	8	24.10				
Cor total	3.432E+005	14				Significant	

Table B.7 ANOVA for effect of feed rate and grinding zone temperature on $L*$ value

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	3.29	6	0.55	3.87	0.0411	0.38	0.54
Feed rate	0.036	2	0.018	0.13	0.8821		
Temperature	3.25	4	0.81	5.74	0.0177		
Residual	1.13	8	0.14				
Cor total	4.42	14				Significant	

Table B.8 ANOVA for effect of feed rate and grinding zone temperature on a^* value

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	0.097	6	0.016	3.35	0.0589	0.069	1.80
Feed rate	0.036	2	0.018	3.72	0.0722		
Temperature	0.061	4	0.015	3.17	0.0773		
Residual	0.039	8	4.830E-003				
Cor total	0.14	14				Not Significant	

Table B.9 ANOVA for effect of feed rate and grinding zone temperature on b^* value

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	0.96	6	0.16	1.82	0.2116	0.30	0.97
Feed rate	0.39	2	0.19	2.21	0.1717		
Temperature	0.57	4	0.14	1.63	0.2581		
Residual	0.70	8	0.088				
Cor total	1.66	14				Not Significant	

Table B.10 ANOVA for effect of feed rate and grinding zone temperature on zingiberene content

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	3.75	6	0.62	89.43	0.0517	0.084	0.27
Feed rate	0.051	2	0.026	3.67	0.0740		
Temperature	3.70	4	0.92	132.31	0.0612		
Residual	0.056	8	6.985E-003				
Cor total	3.80	14				Not Significant	

Table B.11 ANOVA for effect of feed rate and grinding zone temperature on geranial (citral a)

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	0.26	6	0.044	1.47	<0.0001	0.17	0.88
Feed rate	0.094	2	0.047	1.56	0.324		
Temperature	0.17	4	0.043	1.42	0.0008		
Residual	0.24	8	0.030				
Cor total	0.51	14				Significant	

Table B.12 ANOVA for effect of feed rate and grinding zone temperature on neral (citral b)

Source	Sum of Squares	df	Mean	F Value	p-value	S.D	C.V
Model	0.088	6	0.015	1.83	0.0036	0.090	0.24
Feed rate	0.055	2	0.027	3.41	0.0848		
Temperature	0.033	4	8.343E-003	1.04	<0.0001		
Residual	0.064	8	8.053E-003				
Cor total	0.15	14				Significant	

APPENDIX-C

Effect of cryogenic conditions on grinding characteristics

Table C.1. Effect on Specific energy consumption (kJ/kg)

Grinding zone temperature (^oC)	Feed rate (kg/h)		
	8	20	35
	Specific energy consumption (kJ/kg) (Mean ± S.D)		
-10	328.4±2.4	274.9±2.9	223.6±3.8
-40	314.8±3.1	267.2±2.1	219.3±2.4
-70	308.7±2.1	261.2±2.6	214.7±3.1
-100	301.7±2.4	258.1±1.5	210.6±2.9
-130	298.1±2.7	254.7±1.7	204.9±3.1

Table C.2. Effect on time of grinding and particle size

Feed rate (kg/h)	Grinding temperature (^oC)	Time of grinding (sec)	Particle size (mm)
8	-10±5	498	0.259
	-40±5	486	0.253
	-70±5	471	0.249
	-100±5	465	0.245
	-130±5	458	0.24
20	-10±5	210	0.285
	-40±5	207	0.283
	-70±5	200	0.279
	-100±5	191	0.274
	-130±5	187	0.271
35	-10±5	135	0.312
	-40±5	129	0.293

	-70±5	124	0.281
	-100±5	122	0.28
	-130±5	115	0.279

APPENDIX-D

Effect of cryogenic conditions on ginger powder quality

Table D.1 Effect of temperature and feed rate on volatile oil content and oleoresin

Feed rate (kg/h)	Grinding temperature (^{o}C)	Volatile oil yield (ml/100 g)	Oleoresin (%)
8	-10±5	2.9	7.12
	-40±5	2.93	7.24
	-70±5	3.12	7.66
	-100±5	3.15	7.68
	-130±5	3.16	7.76
20	-10±5	2.93	7.08
	-40±5	2.95	7.28
	-70±5	3.12	7.62
	-100±5	3.15	7.67
	-130±5	3.16	7.72
35	-10±5	2.93	7.14
	-40±5	2.96	7.27
	-70±5	3.12	7.61
	-100±5	3.15	7.66
	-130±5	3.16	7.79

Table D.2 Effect of temperature and feed rate on colour values

Feed rate (kg/h)	Grinding temperature (^{o}C)	Hunter colour values		
		*L **	*a**	*b**
8	-10±5	68.66	4.01	30.61
	-40±5	68.8	4.03	30.83
	-70±5	68.99	3.94	30.32
	-100±5	69.99	3.79	29.77
	-130±5	70.24	3.89	30.54
20	-10±5	69.24	3.92	30.62
	-40±5	69.65	3.87	30.72
	-70±5	68.45	3.81	30.41
	-100±5	69.8	3.89	30.21
	-130±5	69.99	3.75	30.85
35	-10±5	69.16	3.89	30.5
	-40±5	69.21	3.87	30.6
	-70±5	69.29	3.8	30.72
	-100±5	69.36	3.88	30.95
	-130±5	70.23	3.64	31.25

APPENDIX-E

Changes in quality of ginger powder during storage
Table E.1 Changes in volatile oil content (ml/100 g)

Packaging material	Month 1	Month 2	Month 3
Al foil	3.16	3.16	3.16
LDPE 400	3.15	3.15	3.14
LDPE200	3.16	3.15	3.13
LDPE100	3.15	3.14	3.13

Table E.2 Changes in moisture content (%w.b.)

Packaging material	Month 1	Month 2	Month 3
Al foil	10.5	10.51	10.52
LDPE 400	10.58	10.61	10.61
LDPE200	10.57	10.59	10.62
LDPE100	10.6	10.64	10.67

Table E. 3 Changes in oleoresin content (%)

Packaging material	Month 1	Month 2	Month 3
Al foil	7.69	7.7	7.56
LDPE 400	7.69	7.65	7.52
LDPE200	7.56	7.59	7.54
LDPE100	7.66	7665	7.68

Table E. 4 Changes in colour values

	Packaging material	Hunter 'L' value	Hunter 'a' value	Hunter 'b' value
Month 1	Al foil	69.8	3.88	30.95
	LDPE 400	69.4	3.89	30.96
	LDPE200	69.8	3.87	30.96
	LDPE100	69.4	3.88	30.94
Month 2	Al foil	69.3	3.89	30.91
	LDPE 400	69.2	3.87	30.92
	LDPE200	69.4	3.99	30.93
	LDPE100	69.7	3.97	30.92
Month 3	Al foil	69.1	3.89	30.89

	LDPE 400	69	3.89	30.82
	LDPE200	68.4	4.02	30.83
	LDPE100	68.2	4.01	30.81

APPENDIX-F

Estimation of Cost of Production of cryoground ginger powder

Capacity of the grinder = 35 kg/h
Working hour perday = 10 h
Total capacity of the unit per day = 350 kg/h
Cost of the grinder (S) = Rs. 3,00,000
Life span of the unit (n) = 15 years
Annual usage (A) = 300 days
Interest rate (i) = 11 % per annum

I. Fixed cost per year

Fixed cost of the grinding unit (C) = $[i(i+1)^n/(i+1)^n +1]*S$
= $[0.11(0.11+1)^{15}/(0.11+1)^{15}+1]*$ 3,00,000
= Rs. 27270

II. Variable cost per year

a. Repair and maintenance of grinder = 2 % of initial cost of the grider
= 2/100* 3,00,000
= Rs.6000

b. Cost of energy

Energy requirement (grinder) = 28.4 kWh / 10 h
Energy requirement for
3 light(120 w/h) and 2 Exhaust (80 w/h) = 2 kWh / 10 h

Total energy requirement = 30.4 kWh / 10 h
Electricity charges = Rs. 5.85 / kWh
Electricity consumption charges = No. of days x Energy/day x Rate
= Rs. 53,352

c) Labour charges

One women labour @ Rs. 400/day = 1 × 400
= Rs. 400
Cost of labour per year = 400 x 300
= R s. 1,20,000

d) Cost of raw materials

Cost of dried ginger = Rs. 300 / kg

Total quantity of dried ginger required per day = 350 kg

Cost of dried ginger for 300 days = 350 x 300 x 300

= Rs. 3,15,00,000

Cost of liquid nitrogen = Rs. 80 per litre

Quantity of liquid nitrogen required per kg = 0.5 L

Total quantity of liquid nitrogen required per day = 175 L

Cost of liquid nitrogen for 300 days = 300 X175X80

= Rs. 42,00,000

Total variable cost ginger powder / year = a+b+c+d

= 6000+53,352+1,20,000+3,15,00,000 +42,00,000

= Rs.35879352

Total cost for the production of ginger powder / year (m) = Total fixed cost + Total variable cost

= 27270+35879352

= Rs. 493500232

Total production of dried ginger / year (n) = 350× 300

= 1,05,000 kg/year

Cost of production of one kg of ginger powder = (m/n)

= 493500232/1,05,000

= **Rs. 470**

OPTIMIZATION OF PROCESS PARAMETERS FOR CRYOGENIC GRINDING OF DRIED GINGER

by
BHAVYA FRANCIS
(2014-18-105)

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Kerala Agricultural University

Department of Food and Agricultural Process Engineering

KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND TECHNOLOGY, TAVANUR - 679573, MALAPPURAM

KERALA, INDIA

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ABSTRACT

The processing of spices requires additional care because they are sensitive to atmospheric parameters like temperature and light. The exposure to temperature during any of the processing stage may lead to their quality deterioration in terms of colour, volatile oil, volatile oil constituents, oleoresin content etc. The grinding of spices at cryogenic temperatures helps in retaining the heat sensitive components in the spices. This study envisages on the cryogenic grinding of dried ginger in order to optimize the grinding conditions, considering the important quality characteristics and compared the cryoground powder with the conventionally ground ginger. The physical properties which affect the grinding process directly or indirectly were also determined. The experiments on cryogenic grinding have been carried out for different feed rates and grinding temperature and the best combination was determined in terms of product quality as well as grinding characteristics. The optimized feed rate and temperature were 35 kg/h and -130 ± 5^{o}C. The lower temperature in the grinding zone solidifies the moisture and oil components in the raw material and makes it brittle, which facilitates the grinding easy and faster thus leads to less energy consumption. The extreme cold condition prevents the volatile oil loss and lead to their higher retention. The comparison of cryoground powder with the powder obtained by conventional method has shown that cryogenic grinding prevented the essential oil and oleoresin loss of ginger considerably and retained its constituents. The colour of cryoground ginger powder was superior and the particle size was finer. The time of grinding and specific energy consumption was found lower in cryogenic grinding. The packaging studies on ginger powder showed that laminated aluminum foil is suitable for long term storage.