ISBN 978-93-84468-20-0
Proceedings of International Conference on Advances in Chemical, Biological & Environmental Engineering (ACBEE)
Singapore March 29-30, 2015, pp. 242-249

Cryogenic Grinding of Cassia

Tridib Kumar Goswami and Bhupendra M Ghodki
Agricultural and Food Engineering Department
IIT Kharagpur – 721302, India

Abstract: *Spices have been in part of our food since centuries, and now become integral part of our life. Cinnamon and Cassia are the bark of evergreen trees and is one of the important spices; however there are multiple species of the trees. In the past, Cinnamon and Cassia have been separated into two distinct species: Cinnamomum verum Presl as Cinnamon and the Cinnamomum cassia Presl, Cinnamomum loureirii Nees, Cinnamomum burmannii Blume species representing Cassia. All four are listed in the Code of Federal Regulations as GRAS. These species are the products that are most common in commercial; trade. Cinnamomum loureirii Nees is one of the major economic species of Cinnamomum belongs to family of Lauraceae and commonly known as Vietnamese or Saigon Cassia and is known for its sweeter flavour and higher volatile oil. Typically its volatile oil varies by quality from 1.5 to 3.0 %, during grinding its amount reduces due to the rise in temperature. For the above reasons Cinnamomum loureirii Nees (Cassia bark) has been selected for cryogenic grinding. Some physical and thermal properties of the Cassia bark were evaluated as a function of moisture content; while mechanical properties were evaluated at moisture content of 11.11 % d.b. and analysis of flavouring components of Cassia bark were also done. And then optimization and validation of product, process and machine parameters, viz., grinding temperature, feed rate, speed of rotor and moisture content of Cassia bark for laboratory scale hammer mill on the basis of responses measured viz., particle size analysis, colour difference, volatile oil content and specific energy consumption for cryogenic grinding of Cassia bark was done using* ***Design-Expert 7.0.0 software. Different experiments were designed using central composite rotatable design (CCRD).*** *Most of the physical and thermal properties of Cassia show linear relationship with moisture content except specific heat, bulk thermal conductivity and bulk thermal diffusivity which follows a second order polynomial relationship. The oil content of Cassia bark was found to be 2.6 ml $(100\ g)^{-1}$ and the chromatogram obtained from Cassia bark essential oil shown a total of 42 flavouring components out of which cinnamaldehyde, (E)- shown the highest peak. The optimum value of product, process and machine parameters viz., feed rate, grinding temperature, rotor speed and moisture content for cryogenic grinding of Cassia bark were predicted as of 2 kg h^{-1}, -90.31 ^{o}C, 1572 rpm and 8.42 % d.b. respectively with desirability level of 0.958. and the predicted values of responses viz., specific energy, particle size, colour difference and volatile oil content of 36.227* ***kJ kg^{-1},*** *60.423 µm, 1.721and 3.0 $(100\ g)^{-1}$ respectively at the predicted optimized parameters. The experimental values for specific energy consumption, particle size, colour difference and volatile oil content obtained were 35.850* ***kJ kg^{-1}****, 62.281 µm, 1.792 and 3.0 ml $(100\ g)^{-1}$ respectively, which validates the predicted optimized parameters.*

Keywords: *Cryogenic grinding, physical, chemical and thermal properties, Cassia, volatile Oil.*

1. Introduction

Spices are one of the important agricultural commodities not only in India, but also throughout the world due to high unit price. Cinnamon and Cassia are the bark of evergreen trees and are one of the important spices; however there are multiple species of the trees. The genus *Cinnamomum* has 250 species and many of them are aromatic and flavouring. The most common spices of the Cinnamon and Cassia are: *Cinnamomum verum* Presl (synonym *Cinnamomum zeylanicum* Blume), *Cinnamomum cassia* Presl, *Cinnamomum loureirii* Nees, *Cinnamomum burmannii* Blume. All four are listed in the Code of Federal Regulations as GRAS. These species are the products that are most common in commercial; trade. Many other species are available, although not to any extent in international trade. In past, Cinnamon and Cassia have been separated into two distinct species: *Cinnamomum verum* Presl as Cinnamon or Ceylon Cinnamon and the *Cinnamomum cassia* Presl, *Cinnamomum loureirii* Nees, *Cinnamomum burmannii* Blume species representing Cassia. Cinnamon and Cassia are reddish brown to tan in colour, depending on the species. The whole spice consists of long slender quills of bark [1]. Ceylon Cinnamon is grown primarily in Sri Lanka, while Cassia may be grown in Indonesia, China, and Vietnam.

Cassia is harvested by peeling off the bark on the trees and allowing it to curl up in quills as it dries (Fig. 1.1). Quills are graded, cut, and sold by size. It should be noted that as the bark curls up, insect can crawl inside. The pieces of whole Cinnamon must therefore be cracked and checked for dead or live insects. The FDA recommended Cinnamon bark contains in average, less than 5 % insect-infected pieces by weight [1].

The physico-chemical characteristics vary widely on variety, agro-climatic conditions existing in the production area, harvest, and post-harvest operations. However, the physicochemical quality remains the ultimate attribute for export requirement of spices as these properties delineate its grade in the market. Thus, the prevailing stringent regulations and quality preferences demand on superior technology for production, processing, packaging and transportation. Thus, cryogenic grinding of spices shows promising features for the promotion of export or for the domestic market. The heat of grinding is very destructive to the volatile oil content of Cinnamon. Cryogenic grinding, however, does retain more volatiles [1]. Cryogenics is the branch of physics and engineering that involves the study of very low temperatures, how to produce these conditions, and the behavior of materials at those temperatures [2]. The quality of Cinnamon is assessed primarily on the basis of its appearance and on the content and aroma/flavour characteristics of the volatile oil.

Various types of milling equipments are in use for processing of food materials. These mills may be categorised as impact or hammer mills, attrition mills, pin mill and air swept mills. Each type of mill produces different produce different particle size characteristics. In all mills, the final particle size produced is directly related to the size and shape of screen opening through which the particle travel and the type of blade used. Most of the mills presently under use can be adopted to apply liquid nitrogen for refrigeration with some modification. Generally, mills that are used for food processing are constructed of stainless steel material which is applicable for low temperatures. Carbon steel should not be subjected to extremely low temperatures for long durations because it cannot withstand such a low temperatures. However it is possible to use liquid nitrogen injection into the mill to just remove the heat being generated by the mill and not produce temperature lower than the fracture point of carbon steel.

Machine and operating parameters, viz., grinding temperature, rotor speed, sieve opening size and number of rotor ribs influence the particle size distribution and specific energy consumption [3-5]. Determination of these parameters is useful for selecting the operating conditions or for designing a grinding system for Cassia.

With cryogenic grinding, the temperature of the products can be as low as -195.6 ^{o}C. But such a low temperature is not required for all spices. In practice, it is regulated anywhere from -195.6 ^{o}C to few degrees below ambient temperatures [6].The temperature to be used is determined by parameters, viz., the final product size, colour required etc. of the product. For example, chili pepper could be ground at -115 ^{o}C, while nutmeg, which has a very high oil content, would require temperature of approximately -157 ^{o}C, where as oregano would require a temperature of approximately -7 ^{o}C.

Considering the above facts, the present study was undertaken with the following objectives.

1. To study some relevant physical, mechanical, and thermal properties of Cassia bark (*Cinnamomum loureirii* Nees) as a function of moisture content.

2. To analyze the flavouring components obtained from Cassia bark.

2. Materials and Methods

The materials and methods for determination of relevant physical properties, force-deformation characteristics under compressive loading, thermal properties and flavour characteristics of Cassia bark, also for optimization of cryogenic grinding of cassia bark have been described here under.

2.1 Raw Material

For the present study, Cassia bark (*Cinnamomum loureirii* Nees L.) was obtained from an importer of Cassia from Siligur, Station Feeder Road Siliguri, West Bengal, India in the month of October 2013, which was sold in the name of split Cinnamon. The sample was imported from Tien Thanx Trading and production Co. Ltd. Village-Dan Phuong District, Hanoi City-Vietnam.

2.2 Moisture Content

The moisture content was determined by Entraiment Method (US ISO 939). The average room temperature throughout the test was 31 ^{o}C with variation of ± 1 ^{o}C. Five levels of moisture content ranging from 6-14 % were selected to examine the effect of moisture content on the the thermo-physical properties as well as for grinding of Cassia bark.

2.3 Oil Content

An essential oil is a concentrated, hydrophobic liquid containing volatile aroma compounds from plants. Essential oils are also known as volatile, ethereal oils or aetherolea, or simply as the oil of the plant from which they were extracted, such as oil of clove. Oil is "essential" in the sense that it carries a distinctive scent, or essence, of the plant. Oil content was determined by hydro-distillation method.

2.4 Freezing Point of Oil

The freezing point was determined by direct cooling of Cassia bark oil by liquid nitrogen [7, 8].

2.4 Physical Properties

The size, sphericity and geometric mean diameter of Cassia bark were determined at the initial moisture content of 11.11 % d.b. Other physical properties like true density, bulk density, porosity and coefficient of static friction at various surfaces, angle of repose were determined at different moisture contents varying from 6 to 14 % d.b. The average room temperature throughout the test was 31 ^{o}C with variation of ± 1 ^{o}C.

2.5 Mechanical Properties

The mechanical properties of Cassia bark such as rupture force and deformation at rupture were investigated.

2.6 Thermal Properties

Determination of thermal properties, like specific heat, bulk thermal conductivity, bulk thermal diffusivity and glass transition temperature of Cassia bark have been described as a function of moisture content in the range of 8-14 % d.b. Five replicates for each thermal property were taken and their average values are reported.

2.7 Experimental Procedure for Cryogenic Grinding of Cassia Bark

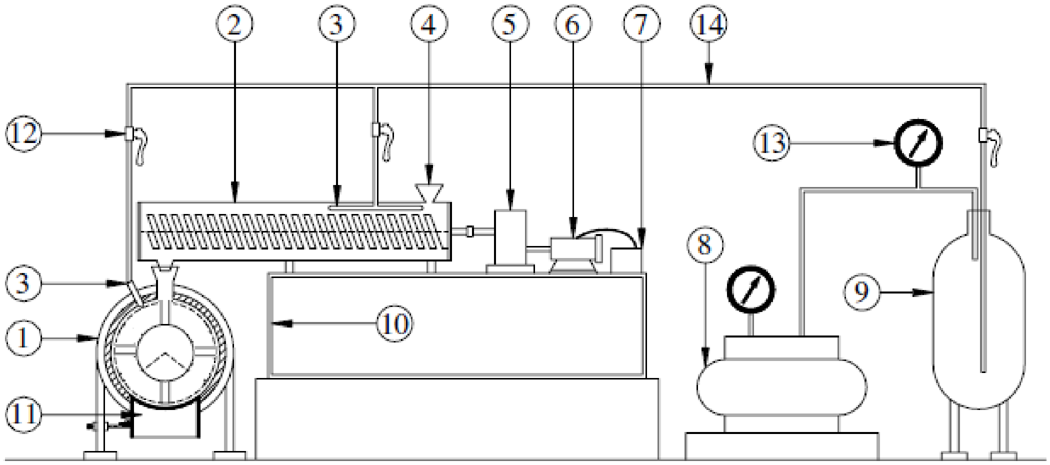
The experimental procedure for optimization of cryogenic grinding of Cassia bark for laboratory scale hammer mill viz., measurement of different parameters, assessment of quality of final product and technique of data analysis are presented and discussed. The average room temperature throughout the test was 31 ^oC with variation of ± 1 ^oC.

For experimentation laboratory scale hammer mill was used in the study as shown in Fig. 2.1, which was fabricated indigenously from Tailibali manufacturers, Kolkata based on the design parameters supplied by IIT Kharagpur. The mill had a rotor of 165×10^{-3} m diameter with 4 number of fixed rotor ribs, with maximum speed of 3000 rpm.

Fig. 2.1: Pictorial view of Hammer mill.

Hammer mill with classifying screen (ASTM 120 mesh) with an opening of 125 µm [9, 10] at outlet was used for size reduction of Cassia bark. Experimental runs were carried out with fixed four ribs of the rotor of the hammer mill. A thermocouple attached to a temperature indicator was placed in the sample collector bag, to carefully monitor and record temperatures of the ground samples just coming from the outlet. The spice samples to be ground under cryogenic conditions were fed through the feed hoper along with liquid nitrogen (LN_2) [11]. For each experimental run, a known fixed quantity of spice (200 g) was fed to the mill. Hammer mill was run from 900 to 2100 rpm at an interval of 300 rpm with the help of a DC motor controller having specifications of 2 hp, 220 V, 8 amp (make, Creative Controls, Mumbai, India). The Cassia bark sample was allowed to enter into the grinder after passing through the precooler by varying the feed rate from 2 to 10 kg h^{-1} at the interval of 2 kg h^{-1}, feeding and grinding temperature from -130 to 30 ^oC at the interval of -40 ^oC with variation of ± 3 ^oC and the moisture content of Cassia bark sample was varied from 6 to 14 % d.b. In case of temperature rise during grinding, the flow rate of liquid nitrogen was increased. The grinding time for each experimental run was 12 min as 85 % of ground Cassia was obtained in 12 min. Energy consumed for each run was noted down from an energy meter (Bentec electrical and electronics, Pvt. Ltd., Kolkata, India) connected to the comminuting mill. The energy meter directly provided energy values for grinding in kWh units with an error of ± 0.1 kWh. The powder was collected in a bag attached to the outlet of the chute and the nitrogen vapour let out. The cryogenic grinding system used in study shown in Fig. 2.2.

The collected powder samples were packed into moisture resistant flexible pouches immediately after grinding. They were sealed properly to check ingress of moisture from the surrounding atmosphere. The samples were stored at -5 ^oC till they were analyzed for particle size, colour difference and volatile oil.



LEGEND: 1. Cryogenic grinder (Hammer mill); 2. Precooler; 3. Liquid nitrogen distributer; 4. Feed hopper; 5. Reduction gear box; 6. Motor; 7. Rectifier; 8. Compressor; 9. Liquid Nitrogen dewar; 10. Supporting frame; 11. Discharge hopper; 12. Flow control Valve; 13. Pressure guage; 14. Liquid Nitrogen transfer line

Fig. 2.2: Schematic diagram of cryogenic grinding system used for Cassia bark.

3. Results and Discussion

The results of some of the physical properties, force-deformation characteristics, thermal properties, flavour characteristics of Cassia bark and optimized condition for cryogenic grinding of Cassia bark are presented.

3.1 Physical Properties of Cassia Bark

The results of the some of the relevant physical properties are presented in this section.

3.1.1 Cassia bark dimensions

The mean dimensions of 100 Cassia barks were taken at 11.11 % moisture content d.b. as shown in Table 3.1.

TABLE 3.1. Dimensions of Cassia bark at a moisture content of 11.11 % d.b.

S.No.	Properties	Value
1	Major axis (length) (mm)	34.79 ± 8.10*
2	Intermediate axis (width) (mm)	1.97 ± 2.89*
3	Minor axis (thickness) (mm)	2.84 ± 1.93*
4	Geometric mean diameter (mm)	11.13 ± 1.95*
5	Sphericity	0.32 ± 0.07*

Note: (a) * Standard deviation from mean; (b) Each value is a mean of 100 test samples

3.2 Mechanical Properties of Cassia Bark

Some of the physical properties viz. length, width, thickness, geometric mean diameter and sphericity of Cassia bark samples were calculated for which the mechanical properties viz. rupture force and deformation at rupture of the samples were determined at the moisture contents of 11.11 % d.b., which are presented in Table 3.2. The length, width and thickness of Cassia bark does not vary much with the position and ranges between 46.938 to 40.892 mm, 17.694 to 13.549 mm and 2.716 to 2.248 mm respectively. The geometric mean diameter and sphericity ranges between 12.993 to 10.824 mm and 0.282 to 0.270 respectively. Fig. 3.1 shows the force-deformation curve of a Cassia bark samples along minor axis at 11.11 % moisture content d.b.

TABLE: 3.2 Effect of compression axis on rupture force and deformation at rupture of Cassia bark at 11.11 % d.b. moisture contents for the specified dimensions

Properties	Position		
	Minor axis (along the thickness)	Intermediate axis (along the width)	Major axis (along the length)
Major axis (length) (mm)	46.938 ± 5.675	40.939 ± 6.230	40.892 ± 5.279
Intermediate axis (width) (mm)	17.694 ± 2.563	14.604 ± 3.952	13.549 ± 2.334
Minor axis (thickness) (mm)	2.702 ± 0.460	2.716 ± 0.554	2.477 ± 0.606
Geometric mean Diameter (mm)	12.993 ± 1.130	10.824 ± 3.338	10.997 ± 1.360
sphericity	0.282 ± 0.046	0.270 ± 0.090	0.272 ± 0.041
Break force (N)	130.512 ± 39.289	109.733 ± 58.664	123.106 ± 86.898
Break deformation (mm)	1.357 ± 0.492	0.908 ± 0.453	1.304 ± 0.429

Each value is a mean of 100 test samples

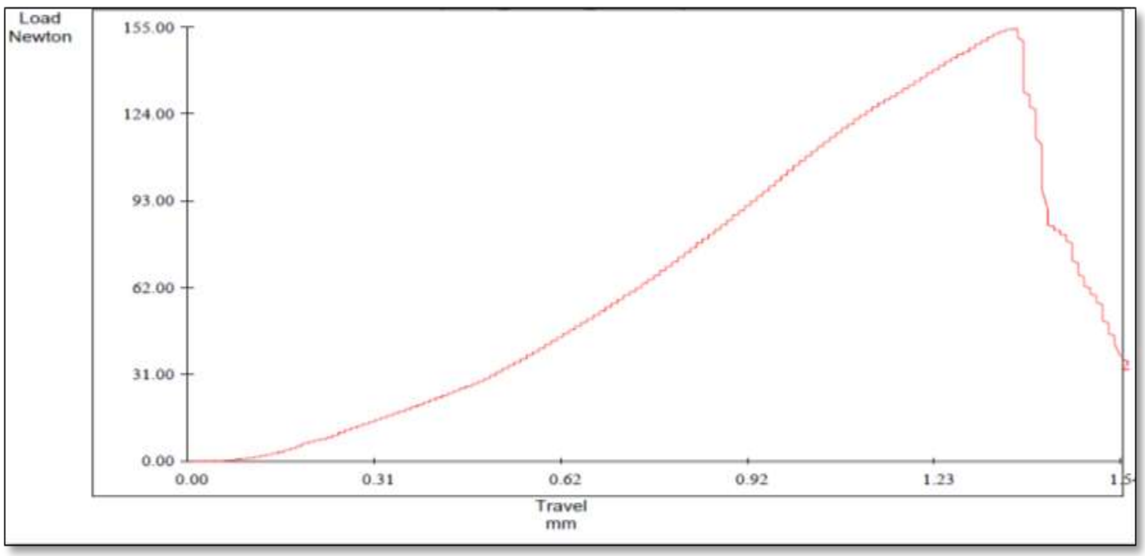


Fig. 3.1: The force-deformation curve of a Cassia bark samples along minor axis at 11.11 % moisture content d.b.

3.3 Thermal Properties of Cassia Bark

The results of some of the relevant thermal properties of the Cassia bark with the variation of moisture content from 8 to 14 % d.b. are presented in this section.

3.3.1 Bulk thermal conductivity

The variation in bulk thermal conductivity with moisture content has been shown in Fig. 3.2. It can be observed that the bulk thermal conductivity increased with the moisture content at 30 ^oC with variation of ± 1 °C and follows linear relationship. The increase is because of the fact that the moisture has higher bulk thermal conductivity than that of air which was present in the void space. A Similar trend was reported for sorghum [12], for pistachios [13], for guna seed [14], for black pepper [15]. and for cashew kernel [15]. The value of bulk thermal conductivity varied from 0.10 to 0.13 W m^{-1} $^oC^{-1}$ with moisture content ranging from 8 to 14 % d.b. The relationship between moisture content and bulk thermal conductivity can be represented by Eqn. (1).

$$k_b = 0.005M + 0.054 \qquad (R^2 = 0.974) \qquad (1)$$

Where, $\alpha_b =$ bulk thermal conductivity of Cassia bark, W m^{-1} $^oC^{-1}$; M = moisture content of cassia bark, d.b.

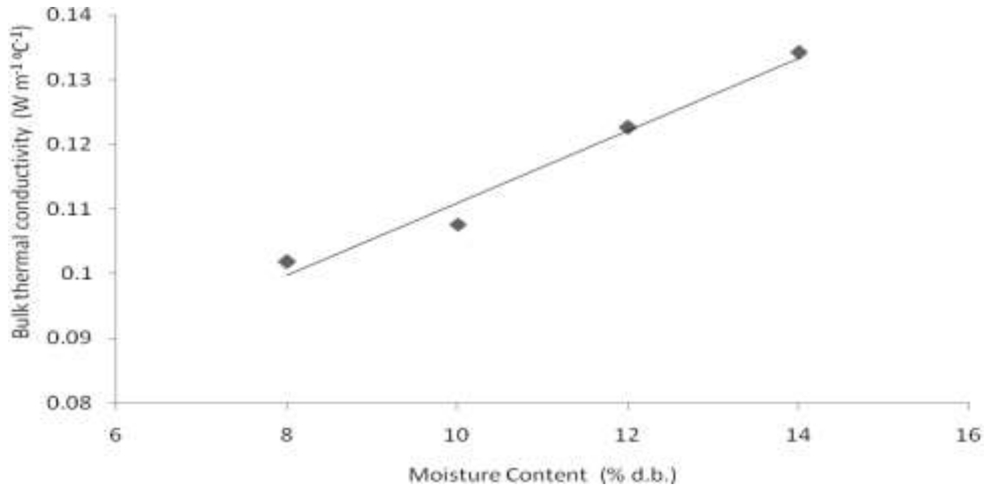


Fig. 3.2: Variation in bulk thermal conductivity of Cassia bark with moisture content.

4. Conclusions

Following conclusions can be drawn from the present study

1. Cassia bark can be successfully ground in laboratory scale hammer mill at the feed rate, grinding temperature, rotor speed and moisture content of 2 kg h^{-1}, -90.31 ^{o}C, 1572 rpm and 8.42 % d.b. respectively to get high quality of Cassia powder at lower energy consumption for 125 micron sieve size.
2. The increase in grinding temperature of Cassia bark in cryogenic range (-130 to -50 ^{o}C) had no significant effect on volatile oil content, whereas, grinding of Cassia in the ambient condition (30 ^{o}C) significantly reduced the volatile oil content.
3. The feed rate and moisture content had no significant effect on volatile oil content of ground Cassia powder; however it reduced with increasing grinding temperature and rotor speed.
4. The moisture content (6 to 14 % d.b.) of Cassia bark had no significant effect on particle size, colour difference and specific energy consumption of ground Cassia powder.
5. As the grinding temperature (-130 to 30 ^{o}C), rotor speed (900 to 2100 rpm) and feed rate (2 to 10 kg h^{-1}) increased the specific energy consumption, particle size and colour difference were also increased.

5. References

[1] D. R. Tainter and A. T. Grenis, *Spices and seasonings: a food technology handbook*, 1st ed. New York, USA: Wiley, 2001.
[2] S. Balasubramanian, M.K. Gupta, and K.K. Singh K. K, “Cryogenics and its application with reference to spice grinding: a review,” *Critical reviews in food science and nutrition*, vol. 52, pp. 781-794. 2012 http://dx.doi.org/10.1080/10408398.2010.509552
[3] D. Landwehr, and M.H. Pahl, “Cold grinding of spices,” *International Journal of Food Technology and Food Process Engineering*, vol. 37, pp. 174-185, 1986.
[4] T. Wolf, and M.H. Pahl, M.H., “Cold grinding of caraway seeds in impact mill,” *International Journal of Technology and Food Process Engineering*, vol. 41, pp. 596-604, 1990.
[5] S. Li, S. Ge, Z. Huang, Q. Wang, H. Zhao, and H. Pan, “Cryogenic grinding technology for traditional Chinese herbal medicine,” *Cryogenics*, vol. 31, pp.136-137, 1991. http://dx.doi.org/10.1016/0011-2275(91)90260-4
[6] J.R. Russo, “Advanced techniques in new spice plant-cryogenic grinding material handling,” *Food Engineering International*, vol. 1, pp. 33-35, 1976.

[7] R.S. Singhal, P.R. Kulkarni, and D.V. Rege, *Handbook of Indices of Food uality and Authenticity* Cambridge, U.K.: Woodhead Publishng Limited, 1997, pp. 386-456. http://dx.doi.org/10.1533/9781855736474.386
[8] M. Meghwal, and T.K. Goswami, T. K., "Thermal properties of black pepper and its volatile oil," *International journal of advanced biotechnology and research*, vol. 2, pp. 334-344, 2011
[9] C.L. Too, Y.A. Yusof, N.L. Chin, R.A. Talib, and M.G. Aziz, "Size reduction of selected spices using knife mill: Experimental investigation and model fitting," *Journal of Food, Agriculture & Environment*, vol. 10, pp.102-106, 2012.
[10] J. Yang, X. Kuang, B. Li, B. Zhou, J. Li, B. CUI, and M. MA, "Study on release mechanism of inhibitory components from cinnamon and clove powders,' *Journal of Food Safety*, vol. 32, pp.189-197, 2012 http://dx.doi.org/10.1111/j.1745-4565.2012.00367.x
[11] R.K. Kumar, "Modification and performance evaluation of indigenous cryogenic grinder," M.Tech. Thesis, Dept. of Agrl. and Food Engg., IIT Kharagpur, Kharagpur, India, 2001.
[12] D.K. Sharma, and T.L. Thompson, "Specific-heat and thermal-conductivity of sorghum," *Transactions of the ASAE* vol. 16, pp.114-7, 1973. http://dx.doi.org/10.13031/2013.37460
[13] M.H. Hsu, J.D. Mannapperuma, and R.P. Singh, "Physical and thermal properties of pistachios," *Journal of Agricultural Engineering Research*, vol. 49, pp. 311-321, 1991. http://dx.doi.org/10.1016/0021-8634(91)80047-I
[14] N.A. Aviara, M.A. Haque, and L.A.O. Ogunjimi, "Thermal properties of guna seed," *International Agrophysics*, vol. 22, pp. 291-297, 2008.
[15] A. Bart-Plange, A. Addo, F. Kumi, and A.K. Piegu, "Some moisture dependent thermal properties of Cashew kernel (*Anarcardium occidentale* L.)," *Australian Journal of Agricultural Engineering*, Vol. 3, pp. 65-69, 2012.