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THERMAL PROPERTIES OF BLACK PEPPER AND ITS VOLATILE OIL

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ABSTRACT
The effect of temperature and moisture content on the thermal properties such as thermal conductivity determined by Thermal Conductivity Meter, specific heat determined by Differential Scanning Calorimeter and bulk thermal diffusivity calculated by standard relations was evaluated. The thermal conductivity was found to be increase 0.1925–0.5986 W m^{-1} $^{o}C^{-1}$, specific heat increased from 1380 to 3980 kJ kg^{-1} K^{-1} with the change in temperature from 20–30 ^{o}C and moisture content (mc) from 5–30% dry basis (db). The thermal diffusivity initially decreased from 16.5 x 10^{-8} to 12.2 x 10^{-8} m^2 s^{-1} with the increase in mc from 5–15% (db), and thereafter increased up to 24 x 10^{-8} m^2 s^{-1} at the mc of 30% (db). The Solidification point, melting point, latent heat and heat of crystallization were found to be -38 ^{o}C, -10 ^{o}C, 225 kJ kg^{-1} and 49 kJ kg^{-1} respectively. Such thermal property evolution data are useful in academia as well as found applicable in designing and fabrications of ambient and cryogenic grinding system for black pepper and other similar commodities.

Keywords: Volatility, Conductivity, Specific Heat, Diffusivity, Density.

INTRODUCTION
A food may be very attractive in look or very nutritious in nature but consumer will not like it if it does not have good aroma and flavor. Black pepper (*Piper nigrum*) is an important and widely used spice throughout the world. It enhances the food by adding flavor and aroma. There are many spices available to consumer, but black paper has commercial value and has many uses besides food enhancement, that is why it is also called as "*Black Gold*" or "*The King of Spices*". It is a flowering vine and belongs to the family *Piperaceae*. Powdered black pepper is used in many different types of meal and food items preparation, to impart the flavor to food dish. Some countries also use it for medicinal purposes to treat and cure many types of illness [1].

Grinding is one of the most common operations used to prepare black pepper powder for consumption and resale. There are many different ambient grinding mills and methods available for this process; such as rotor mill, attrition mill to name a few. However, all operations are performed at high temperatures which create a lot of heat during grinding. Temperature ranging from 40–90 ^{o}C [2] and leads to the loss of flavor, aroma and volatile oil of the ground powder [3]. Cryogenic grinding is a novel and innovative grinding technique. This method helps in retaining good colour, flavor, aroma and volatile oil of the product [4]. The design and fabrication of grinding system or computational fluid dynamics (CFD) modelling needs the statistical data and knowledge about thermal properties governing the heat transfer properties of the material to be ground. Considerations, such as thermal conductivity, specific heat, thermal diffusivity of the commodity to be ground; and, solidification point, melting point, and latent heat of fusion of the oil of the spice commodity to be ground, black pepper in particular.

Despite an extensive literature search no published information was found on thermal properties of black pepper useful for designing, fabricating and modelling the cryogenic grinding system for black pepper. Therefore, this study was performed with the objective to determine some of the major thermal properties such as bulk thermal conductivity, specific heat and bulk thermal diffusivity of the black pepper; and, also to determine the solidification point, melting point, latent heat and heat of crystallization of this commodity.

MATERIALS AND METHODS

Sample Preparation

The black pepper '*Panniyur-1*' was selected for present study. "*Panniyur*" is a name of village in Calicut (Kerala) in which good quality black pepper is cultivated. Based on the village in which this variety of black pepper is cultivated, name of this black pepper variety is kept as "*BLACK PEPPER PANNIYUR-1*". Samples of black peppers were selected and collected from Indian Institute of Spices (IISR), Calicut, Kerala, India. The stones, foreign matters, broken, immature seeds were removed manually from the black pepper sample. The initial moisture content (mc) of the black pepper sample was determined with the help of vacuum oven drying method. In this method sample was heated up to 72 ^{o}C for 24 h until a constant weight was obtained [5-6].

The mc of the black pepper samples were maintained at 5, 10, 15, 20, 25, 30% (db). The initial mc of the black peppers seed samples were 11.7% (db). The samples were dried in vacuum drying oven at 72 ^{o}C (recording mc at every 15 min interval) to achieve ten and five percent mc. The calculated amount of distilled water was added to achieve 15, 20, 25 and 30% mc (db). The black pepper samples were stored in sealed, moisture free and water proof flexible polythene bags. The samples of 15, 20, 25 and 30% mc (db) were kept at five ^{o}C in a refrigerator for one week to allow the moisture to uniformly distribute into the black pepper seeds.

Bulk Thermal Conductivity

The thermal conductivity is the property of material that shows its ability to conduct heat. It is denoted by 'k_b'. It has the unit Watt per meter per degree Celsius (W m^{-1} ^{o}C^{-1}). When the thermal conductivity of a sample of bulk material is determined it is called as bulk thermal conductivity. The bulk thermal conductivity was measured by using Kemtherm QTM-D3, Quick Thermal Conductivity Meter (Kyoto Electronics, Manufacturing (KEM) Co. Ltd, Tokyo, Japan). Probe method of thermal conductivity measurement works based on transient hot wire method. The temperature of the heating wire increases exponentially when the constant power is applied continuously to a thin heating wire which is stretched straight through the rectangular cuboids' sample as shown in the Fig. 1.

As per the prerequisite of the instrument, to get stabilized readings it was run for 10 min before starting the experiments. The black paper samples were filled into the rectangular sample holder having 100 x 100 x 50 mm dimensions. The probe was kept over the flat sample surface to measure the thermal conductivity.

Specific Heat

The specific heat of the black pepper powder was determined using the Differential Scanning Calorimeter **(**DSC) technique by thermal analyser (DSC 204 *Phoenix*, Thermische Analyse, NETZSCH, Germany)**.** The DSC was calibrated with indium and sapphire. To obtain the base line, the DSC was run with an empty crucible by keeping weighed empty two crucibles; one in the reference holder and the other in the sample holder. An empty crucible/pan was used as reference [7]. This run provided base line from which deflection in the heat capacity of sapphire and the sample was determined within the selected range of -150 to 170 ^{o}C. This same technique was used to obtain a standard line using sapphire as a standard sample. For determining the specific heat, black pepper seeds were selected randomly from the whole sample and then crushed with the help of motor-pestle and a portion of the powder obtained was filled in the aluminium crucible (6 mm diameter and 1.5 mm deep) which was covered with an aluminium lid. The sample was brought to the initial temperature of -150 ^{o}C by cooling with liquid nitrogen. It was allowed to equilibrate isothermally and then scanned dynamically at the rate of 10 ^{o}C min^{-1} over the selected temperature range of –150 to 170 ^{o}C. Finally, the weighed five milligram sample of black pepper was taken to run the experiment for specific heat determination by following same technique as that of the reference line [8]. The DSC provided thermogram, in which ordinate shows the heat flow rate mW mg^{-1} as a function of time and temperature [9]. The thermogram is used to evaluate the specific heat of the sample. The thermogram obtained is shown in Fig. 2. With the help of this thermogram using the Proteus Analysis software the desired results such as specific heat, latent heat, solidification point, melting point, and glass transition temperature were evaluated. All experiments were performed in triplicate and the mean values were reported.

Bulk Density

Bulk density is the property of powder, granules, bulk material or other solid food material. It may be defined as the mass of material divided by the total volume they occupy. It is denoted by 'ρ_b' and expressed in kg per cubic meter (kg m^{-3}). The average bulk density of black pepper was determined by using a container of known volume. The container was weighed and weight jotted down. Then container was filled with black pepper and total weight jotted down. The subtraction of weight of container from total weight [7] (i.e., black pepper and container) will give the mass of sample. Now, by using the following Eq. (2) bulk density was calculated Eq. (6) [10].

$$\rho_b = [(\text{mass, kg}) / (\text{volume, m}^3)] \quad (6)$$

Bulk Thermal Diffusivity

The bulk thermal diffusivity of black pepper was calculated from the obtained values of bulk thermal conductivity, specific heat and bulk density of black pepper using the Eq. (8)

$$\alpha_b = \frac{k_b}{(\rho_b c_p)} \quad (8)$$

where, α_b is the bulk thermal diffusivity in m^2 s^{-1}, C_p is the specific heat in kJ kg^{-1} ^{o}C^{-1}, k_b is the thermal conductivity in W m^{-1} ^{o}C^{-1} and ρ_b is the bulk density in kg m^{-3}.

Solidification Point of Oil

The black pepper oil was extracted by the steam distillation technique [6]. Liquid nitrogen was used to determine the solidification point of the oil by the cooling method [2]. Nearly 10 ml of oil was taken in a beaker and liquid nitrogen was directly poured into the oil. The oil was continuously stirred with a glass rod. The solidification point was obtained when solidification of oil started. The temperature of oil was recorded by a temperature indicator (Testo, Germany) having a range of temperature of -190 to 600 with a least count of 0.1 ^{o}C.

Melting Point and Latent Heat of Oil

The melting point and latent heat of fusion were determined by the DSC technique. A five milligram of the oil was weighed in the aluminium crucible for the determination of the melting point. Crucible was sealed by aluminium lid to ensure proper contact between oil and crucible. The black pepper oil was cooled well below its solidification point; then, the temperature was raised at the heating rate of 10 ^{o}C min^{-1}. The heat flow rate into the sample was measured using the DSC thermogram. The melting point was determined as the point of intersection between a base line and the steepest gradient over the selected portion of the curve where it changed linearly. The latent heat of fusion was determined by measuring the area under the thermogram divided by the mass of the sample taken for the melting point determination.

Glass Transition Temperature

In metals like silver or gold there is a sharp melting point i.e., at a particular point of temperature metal changes its state from solid to liquid whereas oil do not have a sharp melting point i.e., they show a range of temperatures in which they exist in a glass like state.

DSC was used to determine glass transition temperatures of the black pepper oil. DSC defines the glass transition temperature as a change in the heat capacity as the solidified oil goes from the glass state to the rubber like viscous state. To obtain glass transition temperature, the sample was first cooled at 10 ^oC min^{-1} rate and then it was heated at the same rate. Glass transition temperature was reported as the onset temperature of the discontinuity/irregularity in the curve of heat flow versus temperature. This is a second order endothermic transition (requires heat to go through the transition) [11].

Heat of Crystallization

The amount of heat that must be removed from one gram of a liquid at its solidification point to freeze it with no change in temperature is known as heat of crystallization [11]. It is a function of temperature history/profile and it also depends on the arrangement of molecules and optical purity [12]. Heat of crystallization was determined using DSC thermogram, the area under the thermogram shows heat of crystallization. It can be used in purification or separation process for oil. The crystallization in frozen dairy desserts brings about an unpleasant sandy mouth feel whereas in oil crystallization may adversely affect other changes like stickiness, caking and loss of volatiles [13].

RESULTS AND DISCUSSIONS

Effect of Moisture Content and Temperature on Thermal Conductivity

Fig. 4 shows the change in the thermal conductivity with respect to moisture content and temperature. It was observed that the thermal conductivity increased with the increase in either of the moisture content or the temperature because increment of both temperature and water in bulk of black pepper material are favourable for the easy conductance of heat. The increase is more significant with the increase in mc compared to increase in temperature within the studied range of mc of 5–30% and temperature of 20–30 ^oC.

Equations were developed with the help of Design Expert software (Trial Version 8) to describe the combined effect of temperature and moisture content on thermal conductivity of black pepper. Eq. (9) gives the polynomial relationship of temperature and moisture content with thermal conductivity (R^2 = 0.98). It is was found from ANOVA (Table 1) that mc has more effect for increase in the bulk thermal conductivity compared to temperature within the studied range of temperature and mc, as P and F value was found to be lower and higher respectively for mc and temperature respectively.

$$k_b = 0.175 - 1.823TE^{-3} - 0.018M + 3.005TME^{-4} + 1.824T^2E^{-4} + 6.853M^2E^{-4} \quad (9)$$

where, T represents the temperatue in ^oC, M represents the moisture content in % (db), and E is the exponential term for scientifically writing of numerical value. Table 1 shows analysis of variance for C_p, k_b, and α_b.

*Significant at 1% level

The variation of thermal conductivity with temperture and mioisture contents are general trend also such trends are in agreement with the study of Kaysoglu et al. (2004) [14] for cereal grains such as barley, wheat, corn and sunflower as these food produces are similar to the black paper in physical and thermal characteristics.

Effect of Bulk Density and Temperature on Thermal Conductivity

It was observed that the thermal conductivity increased with increasing temperature and bulk density of the seed material and results are shown in Fig. 4. The increase is because of the fact that as the bulk density increases the void spaces in the bulk seed materials decreases. Void space decreases this means air retention in the bulk seed material decreases and air being a bad conductor of heat, decrease in quantity of air results in an increase in the bulk thermal conductivity of the seed material. Increase in mc increases the bulk thermal conductivity as moisture has higher thermal conductivity than that of air.

The relationship among the thermal conductivity, moisture content and bulk density is shown in Eq. (10).

$$k_b = -0.925 + 1.937\rho_b E^{-3} + 0.015M \quad (10)$$

In case of mushroom, Srivastav and Datta (1999) [15] reported that thermal conductivity increases sharply with increasing moisture content but the same increases slightly with increasing bulk density. Zuniga and Le-Bail (2009) [16] observed a decreaing trend of thermal conductivity with inceasing porosity which confirms present findings of increasing trend of thermal conductivity with increasing bulk density, since increase in bulk density decreases porosity.

Specific Heat

Specific heat is a function of temperature and moisture content. A linear relationship of specific heat with temperature and moisture content was observed. Fig. 2 shows the typical specific heat determining thermogram for black pepper at 5% mc. Other such thermograms were obtained for determining the specific heat at remaining levels of mc content. As the temperature was raised from 20–30 ^{o}C, specific heat was found to increase from 1380-2800 kJ kg^{-1} ^{o}C^{-1} as shown in Fig.5, whereas the increase in the specific heat with increasing mc from 5–30% (db) was found to be 1380 to 3980 kJ kg^{-1} ^{o}C^{-1} respectively.

Eq. (11) shows the relationship of linear increment of specific heat with increasing mc and temperature among the moisture content, temperature and specific heat.

$$C_p = -497.501 + 32.091T + 291.119M - 3.920TM + 1.638T^2 - 3.529M^2 \quad (11)$$

It is found from ANOVA (Table 1) that mc has more effect for increase in the specific heat of the black pepper compared to the temperature within the studied range of temperature and mc. Also, P and F value was found to be lower and higher respectively for mc and temperature respectively. Muir and Viravanicha (1972) [17] reported that specific heat sharply increases with increase in moisture content as well as temperature and similar trend can be observed from Fig. 5.

Bulk Density

The bulk density of black pepper seed was observed to rise initially from 736–761 kg m^{-3} then decrease from 761–687 kg m^{-3} as the mc level varied 5–30% (db) as shown in Fig. 6.

The relationship between moisture level variation and bulk density can be represented by a regression Equation as shown below:

$$\rho_b = 642.3 + 23.64M - 1.411M^2 + 0.022M^3 \quad (R^2 = 0.894) \quad (12)$$

The initial increase in the bulk density is due to greater increase in mass than volume. On the other hand, as the moisture content was increased beyond 15% (db), volume expansion and pore spaces became proportionally greater which resulted in a decrease of bulk density of black pepper seed. A similar trend of bulk density for other seed materials were reported by many researchers such as for Pistachios by Hsu (1991) [18], for millet by Baryeh (2002a) [19].

Bulk Thermal Diffusivity

The variation of thermal diffusivity with moisture content and temperature can be expressed as shown in the form of Eq.15 :

$$\alpha_b = 58.459 - 3.029T - 1.164M + 0.021TM + 0.056T^2 + 0.025M^2 \quad (15)$$

It can be observed from Fig. 7 that the bulk thermal duffisivity decreases from 16.6 x 10^{-8} to 12.2 x 10^{-8} m^2 s^{-1} with varying mc (db) between 5–15% then after it increases upto 24 x 10^{-8} m^2 s^{-1} with varying mc (db) upto 30%. It is found from ANOVA (Table 1) that mc has more effect for increase in the bulk thermal diffusivity compared to that of temperature within the studied range of temperature and mc, as P and F value is found to be lower and higher respectively for mc and temperature respectively. The initial decrease in bulk thermal diffusivity is due to the fact that intially the bulk density has increased up to 15% mc then it started decreasing and declined upto 30% mc resulting to increase in corresponding thermal deffusivity. Dutta et al. (1988) [20] showed similar relationship between thermal diffusivity and mc for gram cereal.

Solidification Point of Black Pepper Oil

Solidification point of black pepper volatile oil was determined to be -38 ^oC by direct cooling method. Such results are useful for grinding black pepper to its powder because if we have data and idea of the solidification temperature of the black pepper we can adjust the grinding temperature accordingly by application of liquid fluid along with material to be ground. If we can ground the material at solidification temperature then all the volatile oil and aroma content can be retained. Solidification is important aspect for thermal analysis, especially in cryogenic grinding because this grinding is performed at low temperature and determination of solidification at low temperature product is useful for testing related models (Farid, 2001) [21].

Melting Point and Latent Heat of Fusion of Black Pepper Oil

Latent heat of fusion shows the direction of occurrence of thermal process in oil whether it absorbs heat or releases heat. For instance, if oil is heated towards melting and further keep on heating, vaporization of oil starts and the oil absorbs heat and the process is an endothermic one.

As shown in Fig. 8, the point L shows the starting of melting process of oil, the point M shows the peak of melting and the point N is the end of melting process. The melting point (M) was obtained to be -10 ^oC and the corresponding latent heat of fusion was found to be 49.36 kJ kg^{-1}.

Glass Transition Temperature

Glass transition temperature obtained for present thermal treatment of sample may explain, at least roughly favour oil release and loss of aroma strength at various temperatures. It as a function of molecular mass of the substance higher the mass higher is glass transition temperature.

From the DSC thermogram analysis (Fig. 9) the glass transition temperature was found to be onset -0.1 ^oC, mid 0.5 ^oC and end point 1.0 ^oC. The results saw low glass transition temperature which implies that there are week intermolecular interactions in black pepper volatile oil.

Heat of crystallization of black pepper oil

Fig. 10 shows the heat of crystallization obtained for black pepper oil.

Heat of crystallization was found to be 225 kJ kg^{-1} for the heating range of -170 to 150 ^oC. In crystallization process the control of heat is mainly considered as the rate determining step. The same mechanisms that apply to crystallization, also apply to dissolution processes [22]. In the food industry, controlling crystallization is a key factor in controlling quality as it relates to texture of the food material. Some foods require enhancement of crystallization but others may need prevention. Crystallization process plays a great role in lipid processing and their similar applications. In oil, crystallization can be used as a separation process in fat fractionation. Crystallization affects the organoleptic characteristics of black pepper powder or any food stuff. Crystallization temperature helps to know the stability or shelf life of the oil [23].

CONCLUSIONS

The thermal conductivity and specific heat of black pepper are important property were found by Thermal Conductivity Meter and Differential Scanning Meter, and was observed increasing with higher/increasing temperature and moisture content. The decreasing and then subsequent increasing trend of bulk thermal diffusivity shows that up to an optimum level of moisture content and temperature black pepper should be stored in airtight containers, otherwise it may deteriorate rapidly due to the different combination of temperature and moisture content. Solidification point, melting point, latent heat of fusion, glass transition temperature and heat of crystallization of volatile oil were determined; such properties are essential for oil production, processing, storage and quality assessment. These evaluations of thermal properties of black pepper found its application in academia as well as in industry.

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Figures:

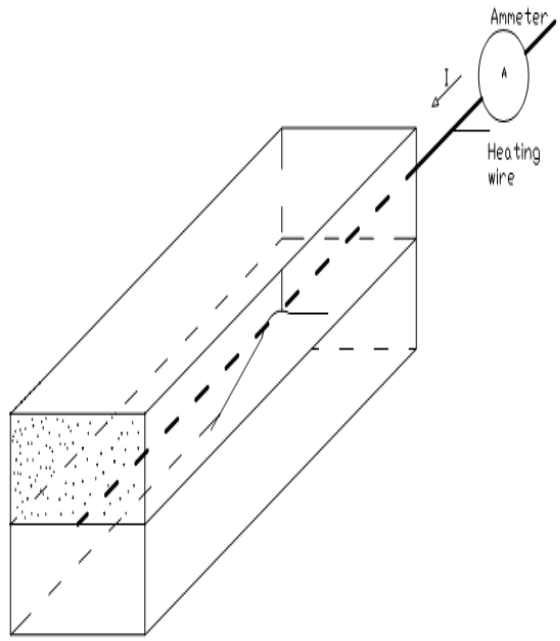
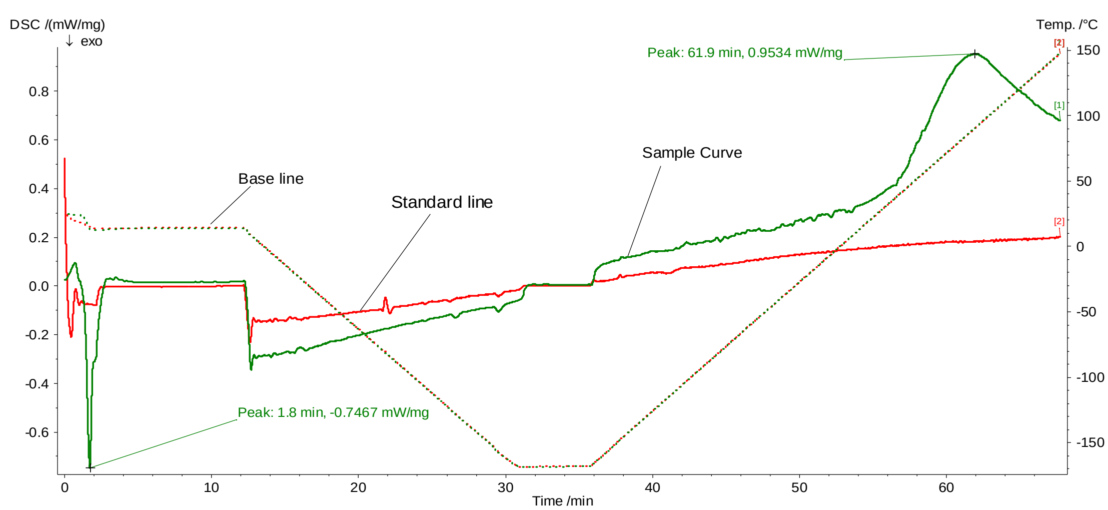


Figure 1 Probe methods for determination of thermal conductivity

Figure 2 A typical thermogram showing the base, standard and sample curve



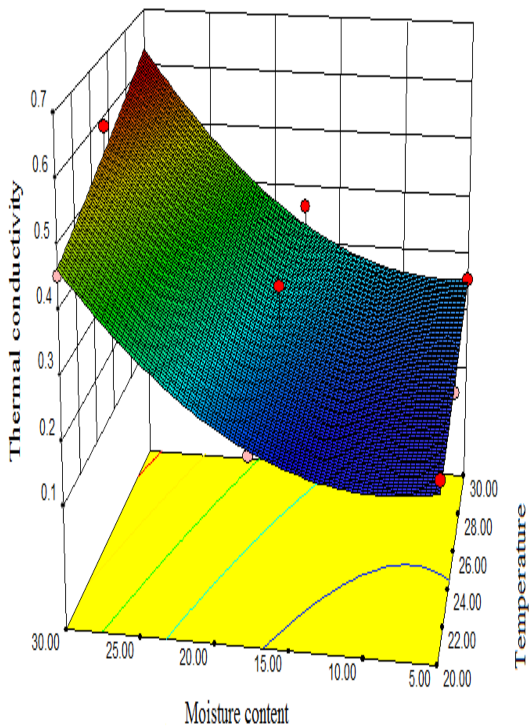


Figure 3 Variation in thermal conductivity with moisture content and temperature

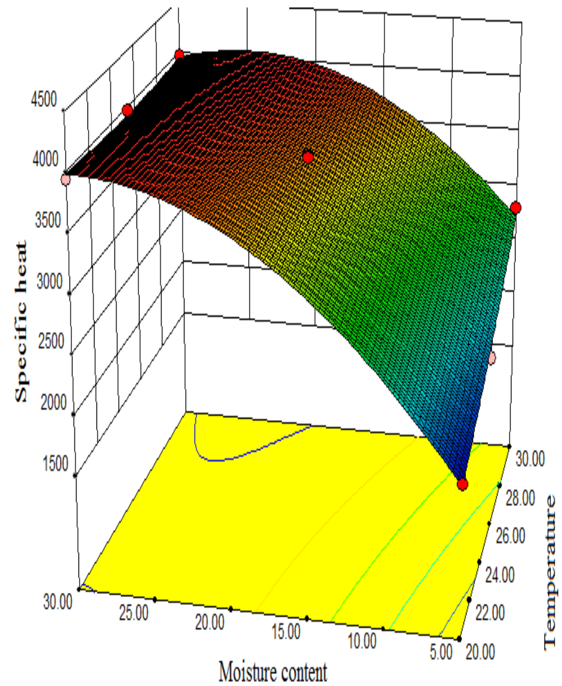


Figure 5 Variation in specific heat with varying temperature and moisture content

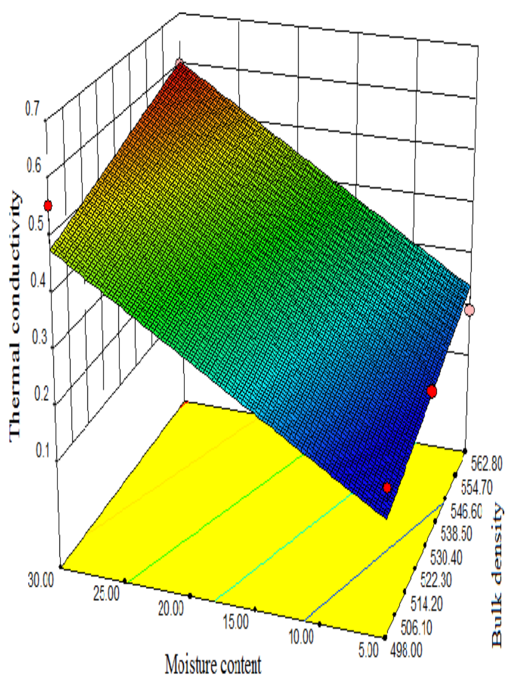


Figure 4 Effect of moisture content and bulk density on thermal conductivity of black pepper.

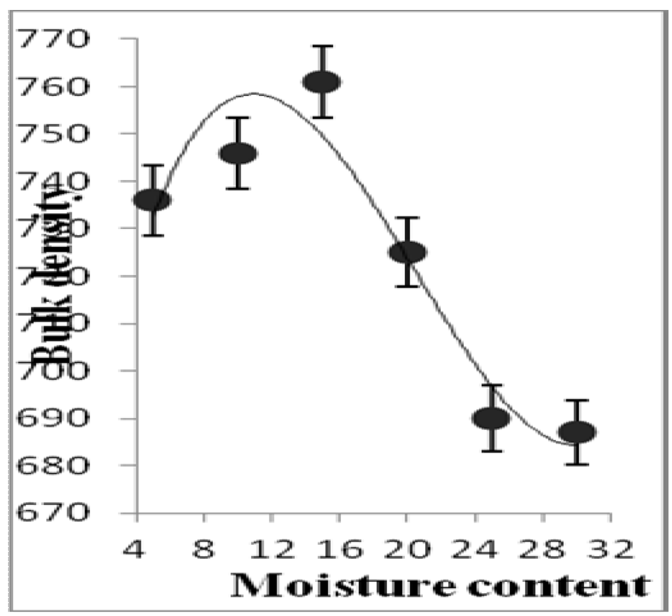


Figure 6 Relationship between bulk density moisture contents

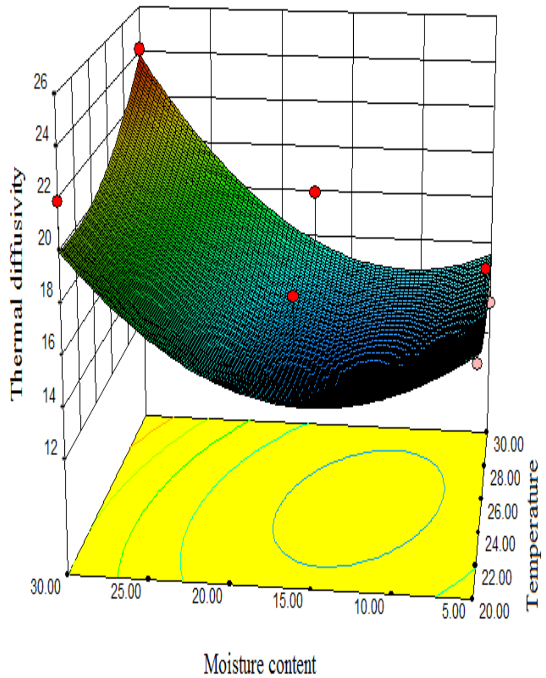


Figure 7 Variation in bulk thermal diffusivity with varying moisture content and temperature

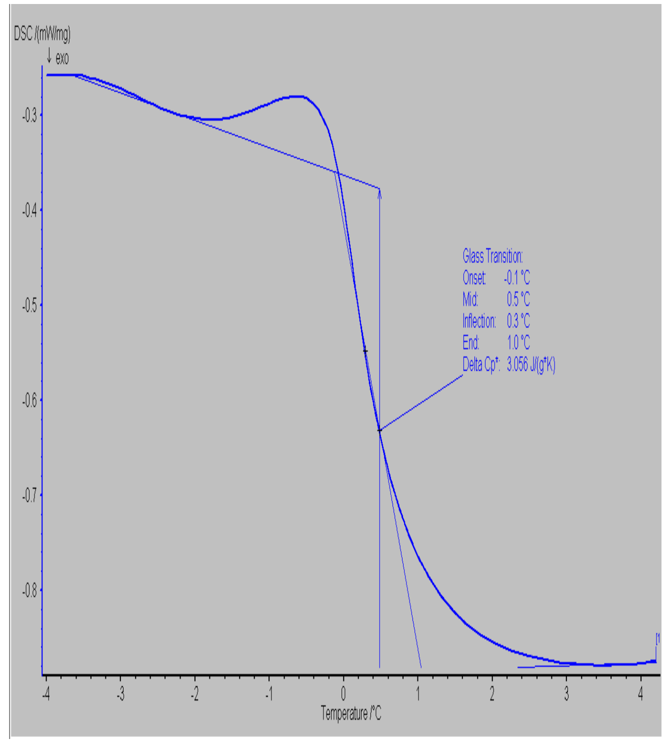


Figure 9 DSC thermograms showing the glass transition temperature of black pepper volatile oil

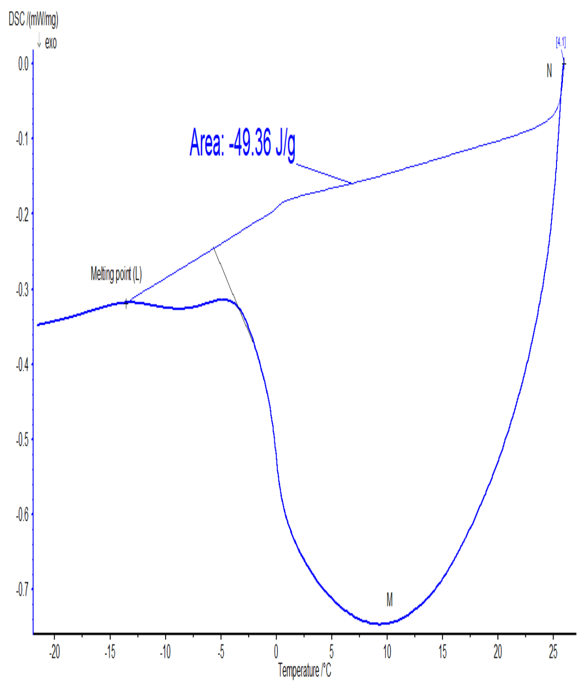


Figure 8 Melting point and latent heat of fusion

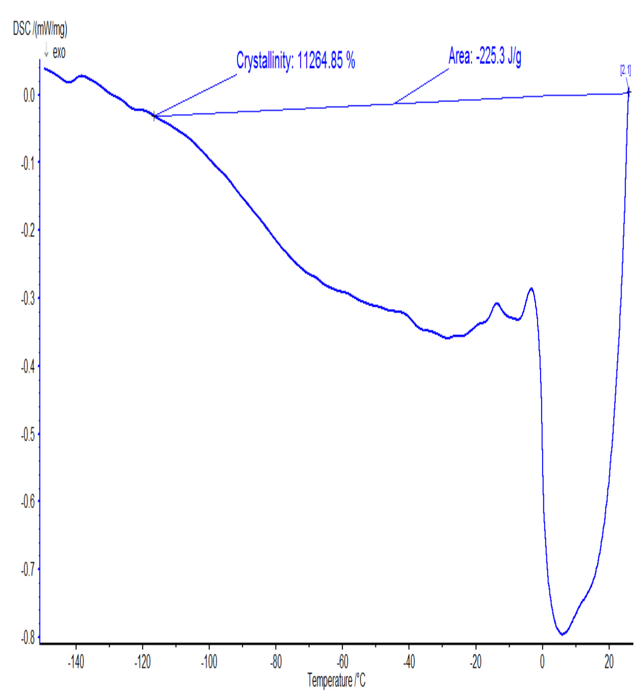


Figure 10 Heat of crystallization

Table 1 Analysis of variance for specific heat, thermal conductivity and thermal diffusivity

Source of variation	**Thermal conductivity**			**Specific heat**			**Thermal diffusivity**		
	DOF	$F_{calculated}$	$P_{caculated}$	DOF	$F_{calculated}$	$P_{caculated}$	DOF	$F_{calculated}$	$P_{calculated}$
Linear term									
Temperature(*T*)	1	6.63*	0.0368*	1	51.63*	0.0002*	1	0.34*	0.5795*
Moisture content (MC)	1	47.49*	0.0002*	1	707*	<0.0001 *	1	7.74*	0.0272*
Quadratic term									
T^2	1	0.016*	0.9026*	1	1.22*	0.3066*	1	1.40*	0.2754*
MC^2	1	8.88*	0.0205*	1	146.25*	<0.0001 *	1	7.21*	0.0313*
Interaction term									
$T \times M$			0.5493*	1	37.38*	0.0005*	1	1.08*	0.3328*
Error	10			8			13		
Total	13			9			15		

*Significant at 1% level