PLANT SCIENCE TODAY
Vol 12(sp3): 01–08
https://doi.org/10.14719/pst.8764

eISSN 2348-1900

RESEARCH ARTICLE

Characterization of thermophysical properties of sorghum (*Sorghum bicolor* (L.) *Moench*) using Differential Scanning Calorimetry (DSC)

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Received: 08 April 2025; Accepted: 14 June 2025; Available online: Version 1.0: 03 October 2025

Cite this article: Anusuya E, Pandiarajan T, Balakrishnan M, Gurusamy K, Nidoni U, Eswari A. Characterization of thermophysical properties of sorghum (*Sorghum bicolor* (L.) *Moench*) using Differential Scanning Calorimetry (DSC). Plant Science Today. 2025;12(sp3):01–08. https:/doi.org/10.14719/pst.8764

Abstract

Sorghum (*S. bicolor* (*L.*) *Moench*) is a significant cereal crop globally, renowned for its nutritional value, drought resistance and versatile applications in food, feed and industrial products. In this study, thermal properties of sorghum were investigated using DSC and Line Heat Source method. Thermal analysis via DSC revealed distinct phase transitions, with the glass transition temperature observed at 70 °C and gelatinization phenomena occurring within the temperature range of 69.5 - 83.3 °C, exhibiting a characteristic endothermic peak at 77 °C. The specific heat capacity was quantified in the range 1.3298 - 1.4522 kJ/kg°C with increasing moisture content and enthalpy was computed as 10.2 kJ/kg. Examination of thermal conductivity as a function of moisture content demonstrated a systematic increase in conductivity values, yielding measurements of 0.094, 0.132, 0.156, 0.189, 0.221, 0.252 W/m°C corresponding to moisture contents of 10, 12, 14,16,18 and 20 % (wet basis) (w.b.) respectively. While thermal diffusivity values ranged from 1.52 x 10^{-4} to 9.61 x 10^{-5} m^2/s. The implementation of DSC methodology facilitated precise characterization of phase transitions and thermal behaviour, generating critical data for various thermal processing operations. These findings constitute significant contributions to the theoretical framework necessary for enhancing process optimization and product quality in sorghum-based applications.

Keywords: glass transition; phase transition; starch properties; thermal analysis; thermal behaviour

Introduction

Sorghum is a distinct cereal crop known for its versatility and nutritional value. It has been originated in northeastern Africa and has been cultivated for the past 5000 years. The remarkable adaptability to diverse agro-climatic condition has led to widespread cultivation across the world. India stands second globally in production of sorghum. Maharashtra leads India's sorghum production followed by Karnataka, Madhya Pradesh, Telengana, Andhra Pradesh and Tamil Nadu (1). Sorghum remains as staple food across many countries in the form of rotis, porridge and fermented products such as kisra, injera. This has also been used in production of commercial food products such as gluten free bakery products, pasta, noodles, ready to eat breakfast cereals, popped and flaked grains. Apart from this it is also used in developing novel foods as in prebiotic formulation, dietary supplements, texturized protein and plant-based meat alternatives.

Sorghum is imbibed with nutritional and functional value such as gluten free, micronutrients (vitamin B), phenols, antioxidants that helps in improving health by lipid regulation, glycemic regulation, gut microbiome regulation and anti-inflammatory activity. Thus, sorghum has an irreplaceable niche in food industry which substantiates the importance of studying the thermophysical property that characterizes the changes in sorghum starch and protein when subjected to heat and pressure (2, 3).

The key thermal properties that are essential for applications such as drying, storage, processing as well as transportation include thermal conductivity, specific heat and thermal diffusivity. Specific heat is the amount of heat required to raise the temperature of a grain by 1 °C. The energy requirement for drying and processing, to maintain temperature stability in storage is based on specific heat capacity. Thermal conductivity is the ability of grains to conduct heat. The heat transfer during drying is influenced by thermal conductivity of grains ensuring uniform drying and cooling along with aeration efficiency during storage. Thermal diffusivity is the rate at which heat spreads through a material. It provides insight on how instantaneously a material can conduct heat relative to its capacity to store heat. The characterization of thermal properties in cereals is fundamental for optimizing processing operations, predicting heat and mass transfer during thermal treatments and designing efficient storage systems. These properties directly influence the quality, safety and efficiency of various processing operations including drying, cooling and thermal treatment.

The methods of determining specific heat include method of mixture, method of guarded plate, adiabatic calorimeter and differential scanning calorimeter. The conventional methods resulted in error during measurement due to thermal gradient while DSC is a dynamic feature that facilitates rapid determination of specific heat as a function of temperature (4 - 6).

DSC is a technique in which the sample is subjected to a controlled temperature and heat flow rate in a substance and reference is measured as a function of temperature. It enables the identification of endothermic and exothermic processes during thermal transition (6). This is a thermo-analytical method that computes the difference in heat flow or variation in temperature between an inert reference and test sample when temperature is raised or lowered. The DSC consists of two cells—one for the sample and one for the reference. Both cells are heated simultaneously and their temperatures are monitored. When a transition occurs in the sample, it requires heat compared to the reference to maintain thermal equilibrium (7). The DSC measures this difference in heat flow, which directly correlates to the energy absorbed or released during the transition. The resulting data enables the determination of various thermal properties such as enthalpy change and heat capacity change (5).

DSC helps in characterizing food components such as determining protein denaturation temperatures and enthalpy which are crucial to understand the thermal stability of proteins, to study starch gelatinization, retrogradation and melting behaviour, which are essential for comprehending the shelf life and texture in starch-based products such as bread and pasta.

The rationale for this study goes beyond the basic evaluation of sorghum's thermal properties to fill crucial gaps in food process engineering and industrial applications. While sorghum is well-known for its nutritional richness and gluten-free benefits, little research has investigated its specific thermal behaviour, particularly in terms of moisture content fluctuations. A thorough study of its thermal transitions such as gelatinization and glass transition is required to optimize processing the processes such as baking, extrusion and drying, making sorghum more competitive in the gluten-free food market.

This study highlights the thermal properties of sorghum grain *viz.,* specific heat, thermal conductivity, thermal diffusivity at different moisture content and characterization of starch properties which comprises of the gelatinization temperature, glass transition temperature and enthalpy (Fig. 1).

Materials and Methods

Raw material

Sorghum (K12) variety was procured from Millet Breeding Station, Tamil Nadu Agricultural University, Coimbatore. The grains were cleaned to remove dirt and other impurities.

Moisture conditioning of samples

A standard method under AOAC was used to determine the moisture content of sorghum grains. After determining the initial moisture content, a precalculated amount of distilled water was sprayed on the samples contained in polythene bags. After proper mixing, the grains were sealed and stored under refrigerated condition for 48 hr. Moisture levels of 10, 12,

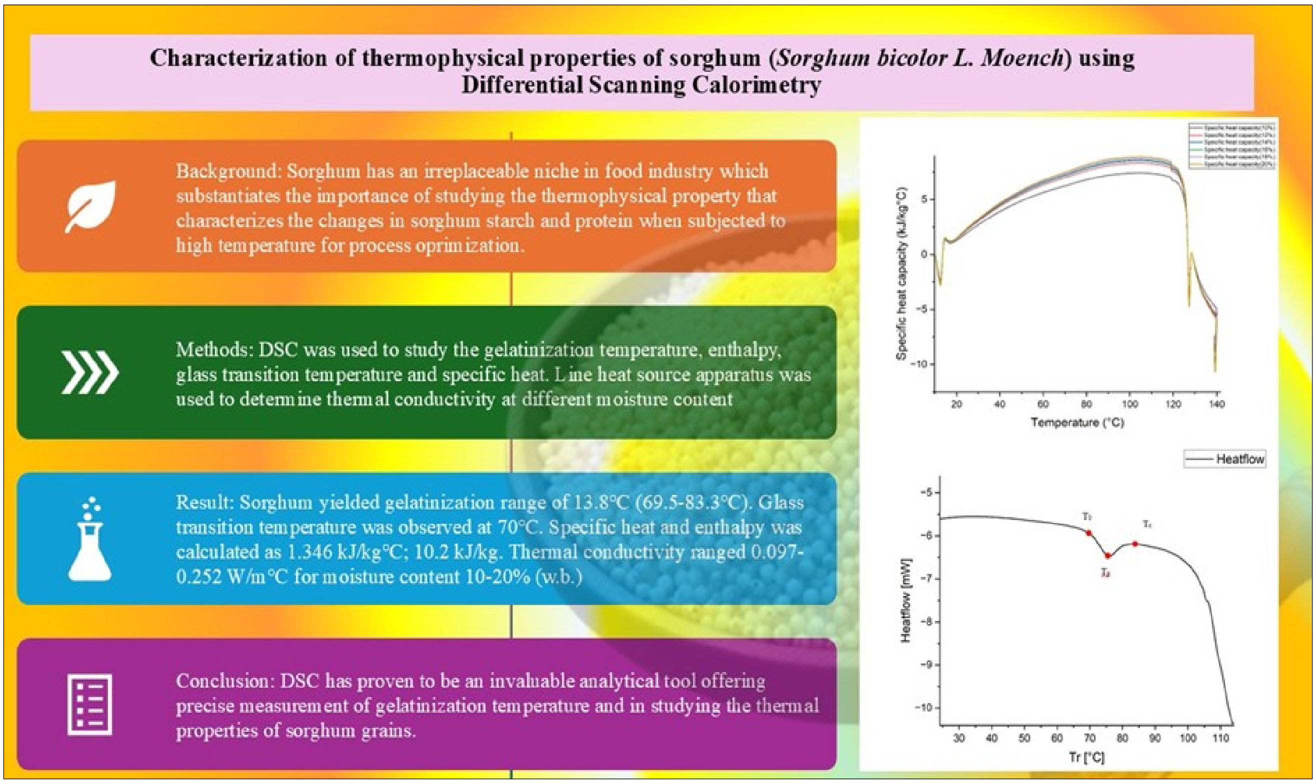

Fig. 1. Highlights of the present study.

14, 16, 18 and 20 % (w.b.) were maintained for the study. The amount of water to be added was calculated by using the Eq. 1 (8):

$$Q = \frac{A(b-a)}{100-b} \quad (1)$$

where Q is the quantity of water to be added (g)in order to achieve the desired moisture content, A indicates the initial weight of sample (g), 'a' refers to initial moisture content present in the sample and 'b' moisture content to be achieved (w.b.) respectively.

Thermal conductivity using line heat source apparatus

Thermal conductivity was determined by transient heat flow method using line heat source apparatus (9). The apparatus comprised of 140 mm diameter PVC cylinder with an insulating particle board in the top and bottom. A nichrome heating element acted as line heat source that is stretched parallel to the axis of the cylinder. A nickel-chromium thermocouple was kept in contact with the heating wire. A variable DC power supply unit and rheostat was used to vary the current flow in the heating element (Fig. 2). The current was turned on and a stopwatch was started while the sample attained a consistent ambient temperature. Up to 10 min, the thermocouple displayed temperature was recorded every 30 sec. The digital voltmeter was used to measure the voltage across a standard resistor to estimate the current with an accuracy of ±0·01 A. A graph was plotted (Fig. 3) against time and temperature rise obtained from thermocouple. dt/dT values obtained from this graph was then plotted against time. The value of time at dt/dT=0 was calculated by extrapolating the resultant straight line and the time correction factor was determined. The time correction factor at 10 % moisture content was found to be 7·5 sec (Fig. 4). Similarly, t_0 for various moisture contents was determined and thermal conductivity values were calculated using Eq. (2):

$$k = \frac{Q}{4\pi\,(T_1 - T_2)} \ln\left(\frac{t_2 - t_0}{t_1 - t_0}\right) \quad (2)$$

Where, k is thermal conductivity (W/m°C), Q is heating rate which is given as the product of square of current (Å) and resistance(Ω), T_1, T_2 (°C) is the temperature at the 1st and 20th second or 2nd and 19th second, t_1, t_2(sec) is the time corresponding to temperature T_1 and $T_2$and $t_0$is the time correction factor respectively.

Specific heat and gelatinization study using DSC

Heat Flux Type DSC (Mettler Toledo) was used to study the specific heat and gelatinization property of sorghum. Samples were made in triplicate by properly weighing 2 mg of flour and dissolving it in deionized water in a pan, to make the water to flour ratio 1:3. The samples were heated from 40 to 120 °C at a heating rate of 10 °C per min. Nitrogen gas flow rate of 20 mL/min was given at an equilibration temperature 5 min at 25 °C (10). ORIGIN PRO software was used to calculate the specific heat, enthalpy and to finding the onset, peak and conclusion temperatures (11). The flow chart illustration of the methodology was provided in Fig. 5.

Calculation of thermal diffusivity

The bulk thermal diffusivity of sorghum grains was calculated from experimentally determined thermal conductivity, specific heat and bulk density using Eq. (3) (12).

$$\alpha = \frac{k}{\rho C_p} \quad (3)$$

Where, α is thermal diffusivity(m^2/s), k is thermal conductivity (W/m °C), ρ is bulk density (kg/m^3) and C_p is specific heat (kJ/kg°C. Bulk density was determined experimentally by taking the grain samples in a beaker of known volume and gently tapped (13).

Statistical analysis

Specific heat data were fitted to linear, polynomial and sigmoidal model using ORIGIN PRO software. The accuracy of the model fit was evaluated based on the coefficient of determination (R^2) which served as primary criterion(11).

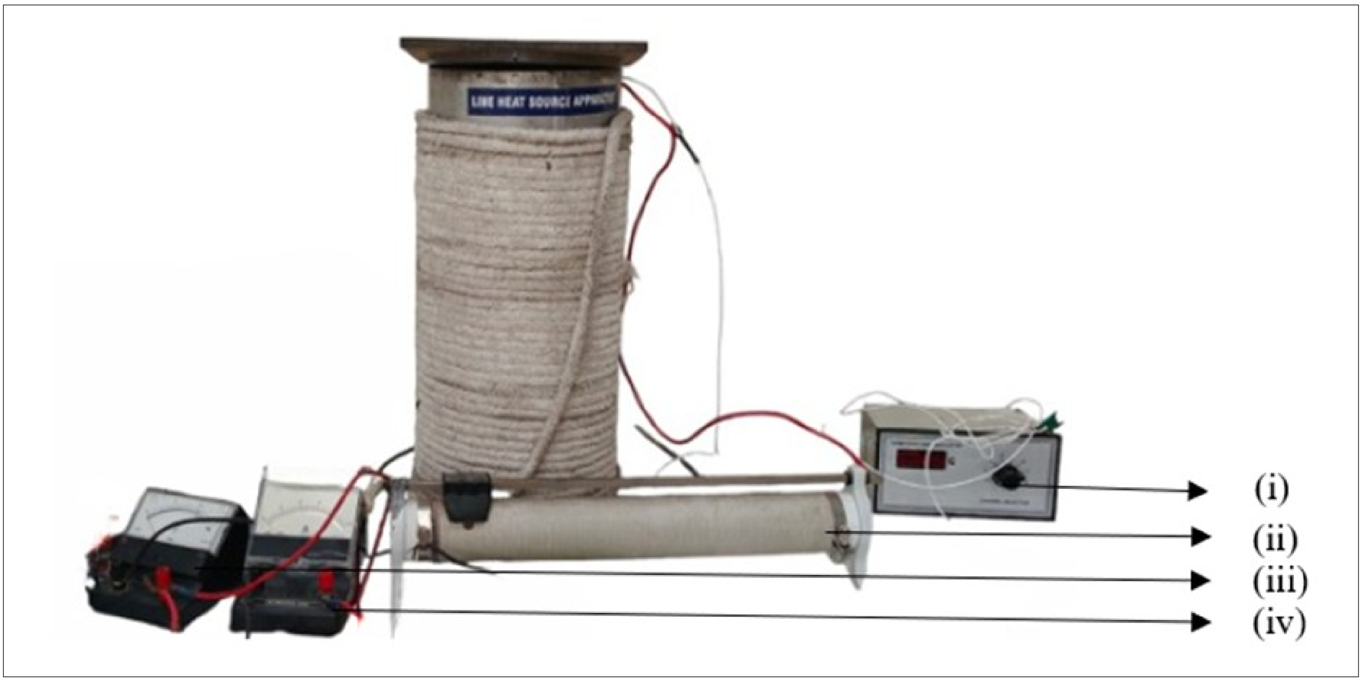

Fig. 2. Line heat source apparatus; (i) temperature indicator; (ii) rheostat; (iii) ammeter; (iv) voltmeter.

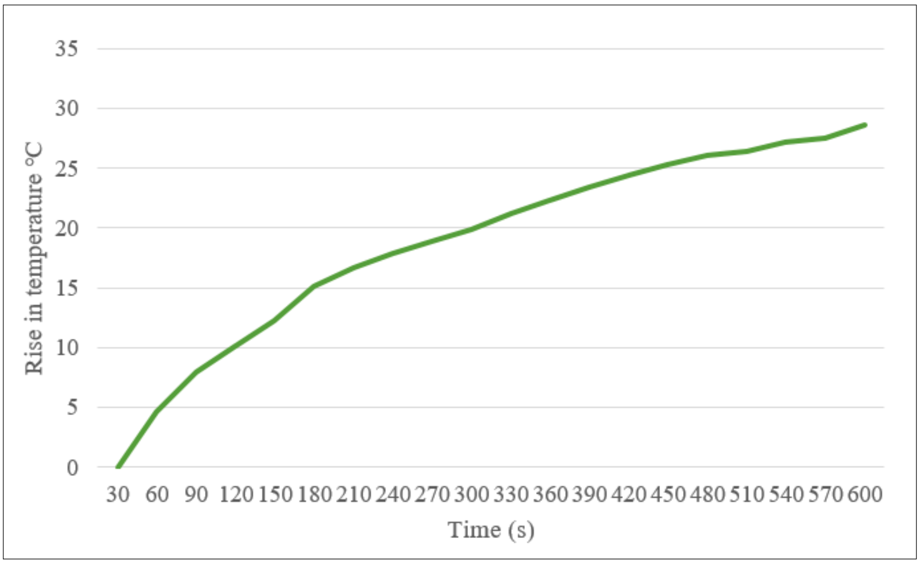

Fig. 3. Time - Temperature plot for sorghum grain. Initial sample temperature: 28.6°C; moisture content 10 % (w.b.); bulk density: 749.14 kg/m3; current: 1.2 A.

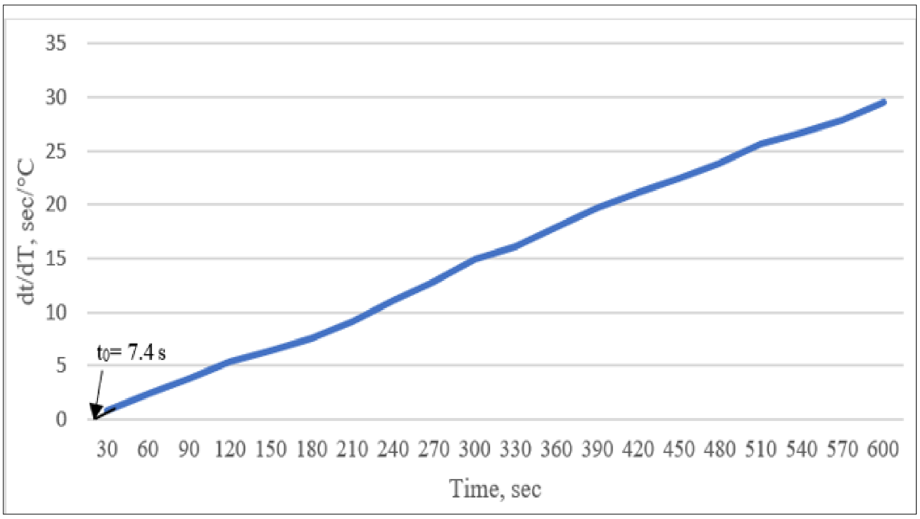

Fig. 4. Plot of dt/dT, sec/°C vs Time (sec) for sorghum grains.

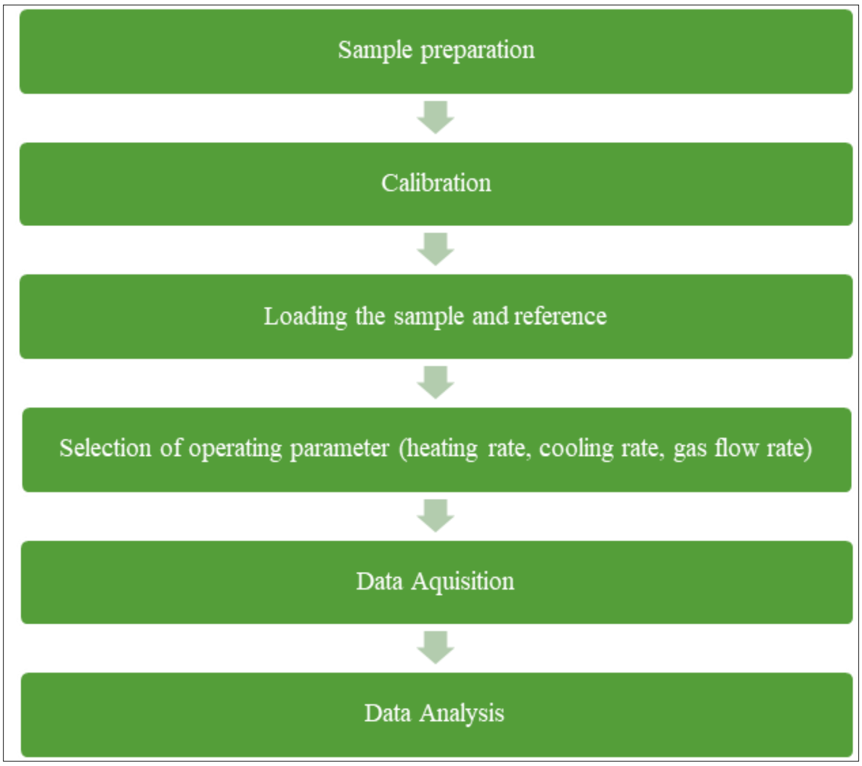

Fig. 5. Methodology for DSC.

Results and Discussion

Thermal conductivity

The experimental results for thermal conductivity of sorghum grains were observed to increase linearly from 0.097 to 0.252 W/m°C with moisture content 10 to 20 % (Fig. 6). Thermal conductivity of grains depends on several key factors such as moisture content, bulk density and temperature. The results were in accordance with the thermal conductivity of other grains; thermal conductivity value was reported to increase linearly with moisture content for wheat (0.181 to 0.202 W/m°C), corn (0.162 to 0.186 W/m°C) (14). This is due to the high thermal conductivity (0.628 W/m °C) of water, which is very much higher than solid and air particles (15, 16). The relationship between moisture content and thermal conductivity was represented using the following Eq. (4):

$$K=0.015583M-0.05933 \quad (4)$$

where K is the thermal conductivity (W/m°C) and M is the moisture content in per cent (w.b.) of sorghum respectively.

Calorimetric Study

DSC plot obtained for the sorghum grains showed endothermic heat flow behaviour where the granular structure responds uniformly to heating. Thermogram indicated the onset (T_o), peak (T_p) and endset or conclusion (T_c) temperature of sorghum grain (Fig. 7). The onset temperature marks the beginning of granule swelling, while peak temperature indicates the maximum gelatinization rate, which is critical for processing conditions. The complete loss of crystallinity concludes the gelatinization process. The thermogram obtained for sorghum grain closely resembled to thermal behaviour reported in previous studies for grains such as rice flour, millet flour(17). The results indicated that sorghum yielded gelatinization range of 13.8°C (69.5-83.3°C). The onset temperature (T_o) which marks the beginning of transition was observed at 69.5°C. The glass transition temperature (T_g) reflects the molecular mobility and shift in physical property; at this temperature the glass state changes to rubbery or melt state. Only above T_g translation motion of the polymer chain is active and resembles a thermodynamic second-order transition as there is sudden increase in specific heat capacity. The temperature in the gelatinization range that stands

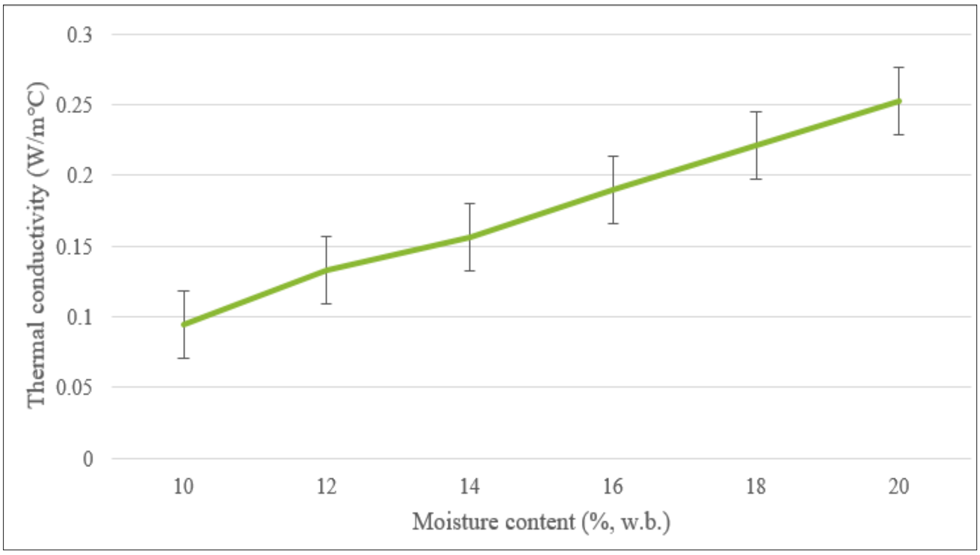

Fig. 6. Plot of moisture content vs thermal conductivity.

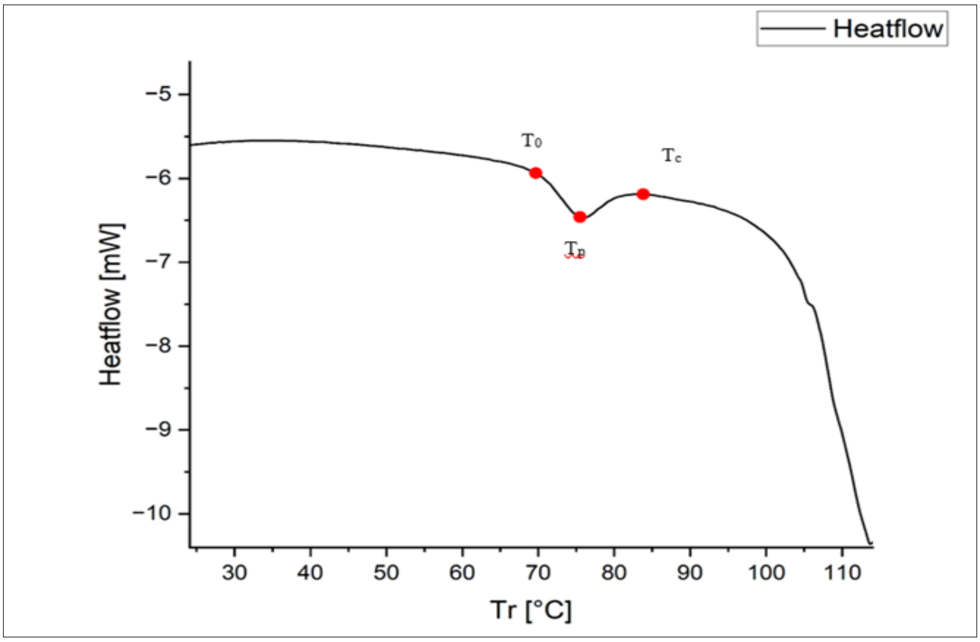

Fig. 7. Thermogram of sorghum.

in transition curve at which the starch molecules changes to melt was observed at 70°C indicated the glass transition temperature. These findings align with previous studies (10) of gelatinization behaviour of millet flours from DSC analysis that revealed distinct thermal properties among different millet. Pearl millet exhibited highest enthalpy change (9.41 kJ/kg) which indicates higher energy requirement for starch gelatinization. The study (18) measured the onset and peak temperatures for gelatinisation of rice flour and starch using differential scanning calorimetry (DSC) as 61.1±0.8 and 67.9 ± 0.8°C respectively. The reported temperatures fall within the expected range for rice starch systems, as documented in previous studies, although slight variations may be observed due to starch type, amylose-to-amylopectin ratio, and presence of interacting solutes.

The enthalpy ΔH was calculated as 10.2 kJ/kg. The enthalpy values of the gelatinized starch indicated the melting of crystallites that were formed during gelatinization and endotherm peak is attributed to melting of gelatinized amylopectin other than amylose. The transition temperature indicates the stability of granular structure against gelatinization (19). Thus, higher gelatinization range depicts more stable amorphous region or lower degree of chain branching. The gelatinization range is affected by the variety and wax content present in the grain; waxy varieties have shorter range when compared to non-waxy starch variety. Sorghum grains have much shorter gelatinization range when compared to other grains such as foxtail, finger millet which illustrated better gelatinization character compared to other grains which in turn provided better starch modification control, reduced processing time and lower energy consumption (20-22).

Specific Heat Capacity

Specific heat capacity is the heat requirement to increase the sample temperature by 1°C. The specific heat capacity was observed to increase linearly with increase in moisture content (Fig. 8). The values were recorded to be in the range of 1.329 - 1.452 kJ/kg°C. This can be explained by the higher heat capacity of water compared to dry matter in grains (23). The results were consistent with those reported by (24) confirming that the specific heat of cereals and legumes increases with rising moisture content and decreases with increasing temperature. In this study, the specific heat values ranged from 1689.1 to 3224.7 J/kgK for the crops investigated, supporting the observed dependency on both moisture content and temperature as previously documented. for specific heat dependence of cereals and legume with moisture content and temperature. For sorghum the best-fitted curve was found to be sigmoidal (logistic) with high correlation coefficient and low p-value (Table 1 and Fig. 9).

Thermal diffusivity

Bulk density which is given as the ratio of weight of millets to the volume of container yielded the values as 749.14, 738.25, 728.15, 715.69, 708.41, 698.25 kg/m^3 at the moisture content 10, 12, 14, 16, 18 and 20% (w.b.) respectively. Thermal diffusivity was observed to increase with increase in moisture content. The value of thermal diffusivity ranged from 1.52 x 10^{-4} m^2/s to 9.61 x 10^{-5} m^2/s. A similar trend was reported for soybean pod as a function of moisture content and temperature (25), for rice grain (26) and for locust beans (27). The relationship between moisture content and thermal diffusivity for sorghum grain is given by Eq. (5)

$$\alpha = 0.141917 \times 10^{-4}M - 0.44333 \times 10^{-4} \quad (5)$$

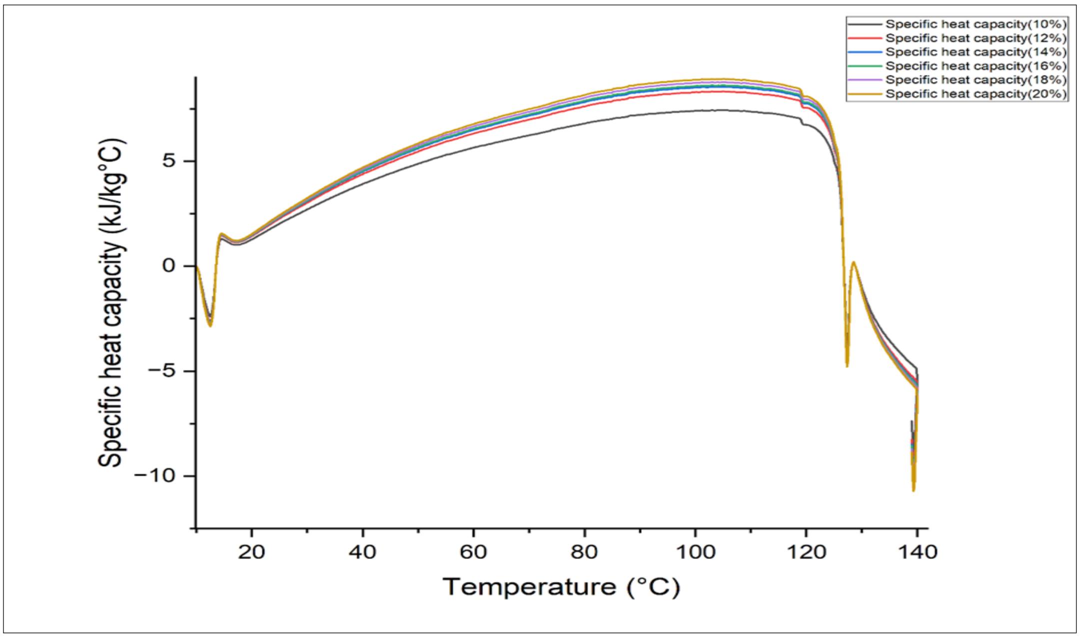

Fig. 8. Plot of specific heat capacity vs temperature at various moisture contents.

Table 1. Equation fitted to specific heat capacity curves

Equation	Model
Linear	0.31+0.07725x; R^2=0.94688 (x=temperature (°C))
Sigmoidal (Logistic)	11.501+(4.486-11.501)/(1+(x/35.492)^1.035; R^2=0.9836
Sigmoidal (Weibull)	7.674-(7.674)*exp(-(0.0271*x)^1.508; R^2=0.9462

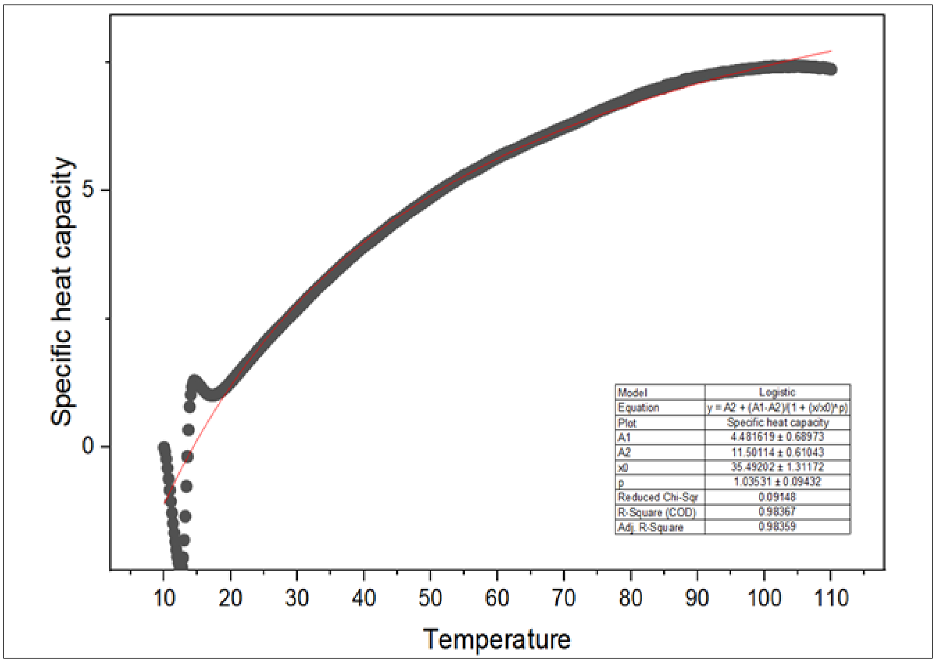

Fig. 9. Best-fitted curve for specific heat capacity of sorghum.

Conclusion

The characterization of sorghum gelatinization and study of thermal properties using DSC provides crucial insight for both research and industrial application. DSC has proven to be an invaluable analytical tool offering precise measurement of gelatinization temperature ranging from (69.5 - 83.3 °C), specific heat value ranged from 1.3298 - 1.4522 kJ/kg °C and enthalpy of 10.2 kJ/kg. Thermal conductivity was determined by line heat source at various moisture contents and the value ranged from 0.097-0.252 W/m °C. Thermal diffusivity values ranged from 1.52 x 10^{-4} m^2/s to 9.61 x 10^{-5} m^2/s for moisture content between 10 - 20 % (w.b.).

Acknowledgements

I would like to express my sincere gratitude to Agricultural Engineering College & Research Institute, Coimbatore for providing access to resources and literature necessary for the completion of this research article. Special thanks to Dr. T. Pandiarajan, Dr. M. Balakrishnan, Dr. K. Gurusamy for their insightful feedback and discussions that helped in shaping the direction of this work.

Authors' contributions

EA and TP worked on the conceptualization. EA did data curation and wrote the original draft. TP carried out supervision, funding acquisition and writing methodology as well as editing. MB contributed to supervision and editing. KG conducted methodology writing along with supervision. UN did supervision and editing. AE performed the statistical analysis.

Compliance with ethical standards

Conflict of interest: On behalf of all authors, the corresponding author states that there is no conflict of interest.

Ethical issues: None

Declaration of generative AI and AI-assisted technologies in the writing process: During the preparation of this work the author(s) used ChatGPT to improve language and readability. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

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