Effects of temperature and particle size on the thermophysical properties of six plant-origin protein supplements

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Abstract: Reasonable design of the parameters of thermal processing such as conditioning and cooling according to formula changes of pelleted feeds has always been a serious challenge for Chinese feed mills and feed equipment manufacturers. Studying the thermophysical properties of different protein feeds under different temperatures and particle sizes will facilitate the equipment design, parameter optimization, and simulation for the thermal processing of pelleted feeds. In this study, the specific heat (C_p), thermal conductivity (k_b), and thermal diffusivity (α) of six plant protein supplements with three particle sizes were determined over a temperature range of 25°C-100°C. The differences in C_p, k_b, and α among different feedstuffs and particle sizes were analyzed and the influences of temperature and particle size on these properties were evaluated. Results showed that the C_p, k_b, and α of all the feedstuffs increased with increasing temperature and varied from 1.622 to 2.417 kJ/(kg·°C), 0.080 to 0.362 W/(m·°C), 6.379×10^{-8} to 21.984×10^{-8} m^2/s, respectively. To rise to the same temperature, the distiller's dried grain with solubles (DDGS) needed to absorb 3% more heat than that required for soybean meal (SBM), while the rest four feedstuffs just needed to absorb 93%-98% heat for SBM. Particle size had no significant effect on C_p for all the feedstuffs (p>0.05). However, descending trends in k_b and α were observed with increasing particle size for a certain feedstuff at the same bulk density. In addition, regression equations with only statistically significant terms were developed to describe C_p, k_b, and α as a function of temperature and particle size for six feedstuffs. The results can provide basic theory and data for the optimization of thermal processing parameters required for the plant-protein ingredient change in compound feed formulations.

Keywords: plant protein supplement, specific heat, thermal conductivity, thermal diffusivity, particle size
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1 Introduction

Protein supplements are one of the most expensive and indispensable feed ingredients[1], which account for 10%-20% of compound feed formulations for livestock. Plant-origin protein sources, mainly including oilseed meals, distillery, and brewery by-products are commonly utilized in livestock diets for the advantages of high yield, great biological values, and relatively low prices. Soybean meal (SBM) ranks first in global oilseed meal consumption with 241 million metric t (70% of total)[2]. It contains about 43%-50% crude protein, which is considered to be the preferred plant-origin protein source due to its excellent palatability, ideal amino acids profile, and high digestibility[3,4]. Rapeseed meal (RSM), having 32%-40% crude protein[5], ranks second (12% of total) in global oilseed meal consumption[2]. Sunflower meal (SFM), containing 28%-30% protein, 25%-27% cellulose, and 11%-13% lignin (dry basis)[6], is considered a potential protein source for ruminants with high-fiber content[7]. Cottonseed meal (CSM) is rich in protein from 30% to 50% in dry matter[8]. Peanut meal (PNM) possesses a high nutritive value with approximately 45%-55% of proteins[9,10], low anti-nutritional factors, and a broad variety of bioactivators[11]. As for distillery and brewery by-products, distiller's dried grains with solubles (DDGS), particularly maize DDGS, are the most widely utilized in China accounting for 9% of total protein feed consumption following SBM and RSM[1].

The continuous increase in the demand for livestock commodities like meat, milk, and eggs, driven by the fast-growing population and rising incomes in developing countries[12], is producing a global shortage of SBM supply. The tight supply and high price of SBM are driving Chinese feed manufacturers to use more economical and local protein sources (DDGS, RSM, CSM, SFM, and PNM) to produce pelleted feeds. However, the modification of feed formulation is a time-consuming process, because the technological parameters of production processes, especially conditioning and cooling (vital thermal-processing processes of transforming the physical state of mixed feed meal), should be adjusted according to the processing properties of feed ingredients. Technical personnel usually cannot effectively adjust conditioning or cooling parameters in practical production due to the lack of comprehensive understanding of the differences and variations in the thermal processing properties of different protein supplements. Consequently, problems such as longer trial-manufacture time, lower pelleting productivity, and poorer consistency of particle quality will occur in the first production after changing the formula. Since protein supplements are required to be

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ground into different particle sizes to produce feed products that are suitable for animals at various growth stages, it is extremely important to investigate the thermal properties of different protein supplements as affected by temperature and particle size for reasonably designing thermal-processing parameters.

The specific heat (C_p), thermal conductivity (k_b), and thermal diffusivity (α) are the most important thermodynamic parameters highly utilized in calculations of heat transfer and energy balance for modeling, simulation, and optimization of thermal processing, like heating, cooling, drying, and storage[13-16]. So far, a considerable amount of experimental investigations explored the thermophysical properties of grains[13,17-19], kernels and seeds[15,20-22], and fruits and vegetables[14,23-25] as affected by moisture content or temperature. In contrast, relatively few researches have been conducted on the thermophysical properties of feed ingredients. The effects of moisture content, temperature, particle size, and whey powder level on C_p of mash feed were investigated and modeled by Kong et al.[26,27], and their results contributed basic data for the establishment of heat-transfer equations for the conditioning process of pelleted feed for weanling pigs. Zielinska[28] reported α, C_p, and k_b of maize/wheat dried distillers grains and dried distillers solubles over the temperature range of 20°C-40°C, however, this range did not cover the thermal processing temperature of pelleted feeds. Researches by Mosqueda et al.[29,30] discussed the effects of condensed distillers' solubles level, drying temperature, and moisture content on k_b and α of wheat DDGS only at room temperature. Kong et al.[31] studied the differences in C_p, k_b, and α of eight energy feedstuffs (four cereal grains and four processed by-products) over the temperature range of 25°C-100°C, which could provide theoretical guidance for the optimization of thermal processing parameters required for the energy ingredient change in compound feed formulations. As far as we know, no published reports have been found detailing the differences in thermal properties among different protein supplements.

In this study, the present research determined the C_p, k_b, and α of six plant-origin protein supplements (SBM, maize DDGS, PNM, SFM, RSM, and CSM) with three particle sizes over a temperature range of 25°C-100°C, which fully covered the thermal processing temperature of pelleted feeds. The aims of this study were 1) to evaluate the differences in the thermal properties among different protein supplements and particle sizes; 2) to model the relationships between the thermal properties and the studied variables.

2 Materials and methods

2.1 Materials

Six protein supplements including extruded SBM, maize DDGS, PNM, SFM, RSM, and CSM were supplied by the Feed Branch of Beijing Shou Nong Animal Husbandry Development Co. Ltd., Beijing, China. The proximate composition of these feedstuffs was determined according to ISO methods. Moisture contents of oilseed meals and DDGS were measured using the methods of ISO 771: 1977 and ISO 6496: 1999, respectively; while for crude fat, using ISO 734: 2015 and ISO 6492: 1999, respectively. Crude protein measurements were performed on the Kjeldahl analyzer (Foss, Kjeltec 2300, Hilleroed, Denmark) following the method of ISO 5983-1:2005. Crude ash and fiber were estimated by the methods of ISO 5984: 2002 and ISO 6865: 2000, respectively.

2.2 Sample treatment

The raw protein supplements were ground using a mini-mill (Hongda 15B, Jiangyin, China) to pass through sieves of three aperture sizes (1.5, 2.0, and 2.5 mm) to form feed meals with three particle sizes. The moisture content of all meal samples was conditioned to 11% in order to eliminate the influence of moisture content on the thermal properties. A predetermined quantity of distilled water was sprayed on known-mass meals with an initial moisture content of less than 11%. Meals with a higher initial moisture content were dried in a hot air oven at 30°C to reduce the moisture to 11%. All meals were stirred and rotated in a mixer for 15 min before being packed in sealed polyethylene bags. The bags were kept in a refrigerator at 4°C for one week with 1 min well-shaking every 6 h to enable the moisture to be distributed uniformly throughout the samples. The experimental moisture contents of feed samples are summarized in Table A1 (in Appendix).

2.3 Particle size (geometric mean diameter, d_{gw})

A sieving method (ANSI/ASAE S319.4 FEB2008) was applied to determine mean particle sizes of protein feed meals and the results were expressed as geometric mean diameters. About (100.00±0.01) g meal sample was weighed and poured into a set of woven wire mesh sieves (Endecotts, ISO3310, London, UK), which consists of 13 sieves and a pan with aperture sizes of 3350, 2360, 1700, 1180, 850, 600, 425, 300, 212, 150, 106, 75, 53, and 45 μm from top to bottom. The sieves were shaken for 15 min on a sieve shaker (Endecotts, OCTAGON 200, London, UK) until the mass of feed meal on any one sieve was consistent. The geometric mean diameter of the feed meal is calculated based on the mass of feed meal left on each sieve using the following equation:

$$d_{gw} = \log^{-1}\left[\frac{\sum_{i=1}^{n}\left(W_i \log \overline{d}_i\right)}{\sum_{i=1}^{n} W_i}\right] \quad (1)$$

where, d_{gw} is the geometric mean diameter of particles by mass, μm; d_i is the nominal sieve aperture size of the ith sieve, μm; d_{i+1} is the nominal sieve aperture size in the next larger than i^{th} sieve, μm; $\overline{d}_i = \sqrt{d_i \times d_{i+1}}$ is the geometric mean diameter of particles on i^{th} sieve, μm; W_i is the mass on i^{th} sieve, g; n is the number of sieves (including pan). Each sample was performed in triplicate and the mean particle size was obtained.

2.4 Specific heat (C_p)

The measurements of C_p of the protein supplements were carried out using a differential scanning calorimetry (DSC) (Netzsch, DSC 214 Polyma, Selb, Germany) technique. The DSC thermogram records the heat flow rate of a sample as a function of temperature[32]. The C_p of a testing sample is evaluated by the difference in a signal of heat flow rate between the testing sample and a reference of known C_p. The device utilizes nitrogen as cleaning and shielding gases with a normal flow rate of 40 and 60 mL/min, respectively. Temperature and sensitivity correction for the device are performed using six certified reference materials ($C_{10}H_{16}$, In, Sn, Bi, Zn, CsCl) before sample tests. To obtain the C_p of a feed sample, it was necessary to get a baseline by placing empty crucibles in both reference and sample holders and scanning at the rate of 10°C/min over the selected temperature range of 5°C-120°C. Then the same procedure was duplicated with a slice of standard sapphire (38 mg) and a weighed sample of feed meal (15-18 mg) to acquire a sapphire curve and a sample curve, respectively. The specific heat of the feed sample at any testing temperature is calculated using the following equation:

$$C_p = C_{pstd} \times \frac{DSC_s - DSC_b}{DSC_{std} - DSC_b} \times \frac{m_{std}}{m_s} \quad (2)$$

where, C_p and C_{pstd} are the specific heat of the sample and sapphire (kJ/(kg·°C)), respectively; m_s and m_{std} are the mass of the sample and sapphire (mg), respectively; DSC_s, DSC_{std}, and DSC_b are the DSC record value of heat flow for the sample, sapphire and baseline, mW, respectively. The results are reported as the mean of three replications.

2.5 Thermal conductivity (k_b)

The k_b of protein supplements was determined by KD2 Pro thermal properties analyzer (Decagon Devices, Inc., Pullman, USA) based on the line heat source theory of unstable state heat transfer. The theory describes the increase in temperature of an infinite homogeneous medium (initially isothermal and in equilibrium with its surroundings) caused by a line heat source of constant strength and negligible diameter[32,33]. The heat flow in the medium from the line source can be expressed by the following equation[34]:

$$\frac{\partial T}{\partial t} = \alpha\left(\frac{\partial^2 T}{\partial r^2} + \frac{1}{r}\cdot\frac{\partial T}{\partial r}\right) \tag{3}$$

where, T is the temperature at radius r, °C; r is the radius from the heat source, m; t is the time since the probe energized, s; α is the thermal diffusivity, m²/s. The solution for T is given by[35]

$$T = \frac{q}{2\pi k_b}\left[C - \ln(rn) + \frac{(rn)^2}{2} - \frac{(rn)^4}{8} + \cdots\right] \tag{4}$$

where, q is the heat input, W/m; k_b is the thermal conductivity, W/(m·°C); $n = 0.5(\alpha t)^{(-1/2)}$ (m^{-1}); C is a constant. If rn<0.16 (when r is sufficiently small and t is long enough), T can be expressed by the first two terms of the above infinite series as:

$$T = \frac{q}{2\pi k_b}[C - \ln(rn)] \tag{5}$$

Then the temperature variation between the times t_1 and t_2 can be expressed as:

$$T_2 - T_1 = \frac{q}{4\pi k_b}\ln\frac{t_2}{t_1} \tag{6}$$

Finally, k_b can be calculated as follows:

$$k_b = \frac{q\ln(t_2/t_1)}{4\pi(T_2 - T_1)} \tag{7}$$

The KS-1 probe (1.3 mm diameter, 60 mm length, and 0.02 to 2.00 W/(m·°C) measuring range) of the KD2 Pro analyzer was calibrated with glycerol and distilled water to ensure that it was within the range of initial accuracy before the sample test. A quartz glass cylinder of known mass and volume (50 mm internal diameter, 100 mm height) was filled by adding the feed meal 5 times with several light taps each time for a more homogenous density. Subsequently, the cylinder was weighed and sealed. The bulk density of the same protein supplement was kept constant over three particle sizes. According to the mass of the feed meal (m) and the volume (V) of the cylinder, the bulk density (ρ_b) of the feed meal can be calculated (ρ_b=m/V).

The sealed cylinder was placed in an electro thermostatic oil bath (Guohua, HH-S, Changzhou, China) with a temperature accuracy of ±0.1°C, then put a heavy weight on top to prevent volume expansion of the protein supplement during heating. The feed sample was heated for an hour to achieve thermal equilibrium at expected temperatures[15]. Then the whole probe was inserted vertically into the middle of the feed meal, and the measurement began when the temperature of the probe reached the same as the feed meal. Each k_b value was acquired from the average of three replicates.

2.6 Thermal diffusivity (α)

The thermal diffusivity of the protein supplements can be calculated from experimentally measured values of thermal conductivity, bulk density, and specific heat using the following equation:

$$\alpha = \frac{k_b}{\rho_b C_p} \tag{8}$$

where, C_p is specific heat, kJ/(kg·°C); ρ_b is the bulk density, kg/m³. The probable uncertainty of the calculated α (ω_α) is evaluated using uncertainties of k_b, C_p, and ρ_b (ω_k, ω_c, and ω_ρ) associated with measured k_b, ρ_b, and C_p as[15]:

$$\omega_\alpha = \left\{\left[\frac{\partial\left[\alpha(k_b,C_p,\rho_b)\right]}{\partial k_b}\omega_k\right]^2 + \left[\frac{\partial\left[\alpha(k_b,C_p,\rho_b)\right]}{\partial C_p}\omega_c\right]^2 + \left[\frac{\partial\left[\alpha(k_b,C_p,\rho_b)\right]}{\partial \rho_b}\omega_\rho\right]^2\right\}^{1/2} \tag{9}$$

Substituting Equation (8) into Equation (9) yields:

$$\omega_\alpha = \left[\left(\frac{1}{\rho_b C_p}\omega_k\right)^2 + \left(\frac{k_b}{\rho_b C_p^2}\omega_c\right)^2 + \left(\frac{k_b}{\rho_b^2 C_p}\omega_\rho\right)^2\right]^{1/2} \tag{10}$$

where, ω_k, ω_c, and ω_ρ are calculated from repeated measurements of k_b, C_p, and ρ_b at the 95% confidence limit.

2.7 Statistical analysis

The data were analyzed by One-way ANOVA with Duncan's multiple-range test at a 95% confidence level using SPSS 22.0 statistics software (SPSS Inc., Chicago, IL, USA). Linear and nonlinear regressions were conducted using MATLAB R2019a software (The MathWorks, Inc., Natick, USA) to model the thermal properties of protein supplements as functions of temperature and particle size. The final regression equations were obtained with only statistically significant terms. The best model was selected based on the R^2 value, root mean square error (RMSE), and mean relative percent error (e). The model with the lowest RMSE, e, and the highest R^2 is selected as the optimal model. RMSE and e were calculated by Equations (11) and (12), respectively.

$$\mathrm{RMSE} = \sqrt{\frac{1}{n}\sum_{i=1}^{n}\left(y_{exp} - y_{pred}\right)^2} \tag{11}$$

$$e = \frac{1}{n}\sum_{i=1}^{n}\frac{\left|y_{exp} - y_{pred}\right|}{y_{exp}} \times 100\% \tag{12}$$

where, y_{exp} is the experimental value; y_{pred} is the predicted value; n is the number of the experimental data.

3 Results and discussion

3.1 Proximate composition and particle size

The results of the proximate composition (wet basis) of the raw protein supplements (without sample treatment) are listed in Table 1. The crude protein content of six feedstuffs varied in a wide range of 26.74%-48.92% in which the highest value belonged to PNM and the lowest belonged to SFM. The initial moisture content ranged from 10.94% (SFM) to 11.56% (SBM), presenting little difference. Significant differences were observed in fat and fiber contents among six feedstuffs (p<0.05). RSM contained the highest crude ash content at 7.58%, and DDGS had the lowest at 3.71%.

The mean particle sizes of the protein supplements being ground through three sieves with aperture sizes of 1.5, 2.0, and 2.5 mm are listed in Table 2. It can be obviously seen that the particle size

of the protein feedstuff increased significantly with the increase in aperture size (p<0.05). The particle sizes of feed meals through 2.0-mm sieve were roughly 1.1 times the sizes of those through 1.5-mm sieve, and the same results were observed when 2.5-mm compared to 2.0-mm. Moreover, there were significant differences in particle sizes between different protein supplements (p<0.05). SBM had the largest particle size, i.e. 370.62, 412.93, and 483.70 μm corresponding to 1.5-, 2.0-, and 2.5-mm sieve, respectively. While RSM obtained the smallest particle size, i.e., 187.38, 191.67, and 204.37 μm corresponding to 1.5-, 2.0-, and 2.5-mm sieve, respectively. For the same grinder with the same parameters, the dramatic differences in particle size among six protein supplements might be attributed to the differences in component, original dimension, shape, texture, and hardness[31].

Table 1 Proximate composition of six protein supplements (% wet basis)

Proximate composition	Soybean meal (SBM, extruded sol.)	Distillers dried grains with solubles (DDGS)	Peanut meal (PNM, exp.)	Sunflower meal (SFM, exp.)	Rapeseed meal (RSM, sol.)	Cottonseed meal (CSM, sol.)
Sample image						
Moisture	11.56±0.03^{a}	11.44±0.03^{b}	11.40±0.04^{bc}	10.94±0.04^{d}	11.32±0.02^{c}	11.47±0.05^{b}
Protein	43.82±0.06^{c}	26.78±0.09^{e}	48.92±0.11^{a}	26.74±0.07^{e}	36.60±0.03^{d}	46.59±0.15^{b}
Fat	1.76±0.06^{d}	9.85±0.19^{a}	4.62±0.16^{c}	8.28±0.05^{b}	0.80±0.04^{e}	0.31±0.01^{f}
Ash	5.82±0.12^{c}	3.71±0.12^{e}	4.37±0.01^{d}	4.40±0.04^{d}	7.58±0.00^{a}	6.39±0.07^{b}
Fiber	5.94±0.17^{c}	7.44±0.06^{d}	4.49±0.09^{f}	33.87±0.18^{a}	13.87±0.05^{b}	13.57±0.06^{c}

Note: Results are expressed as means±standard deviation. Values not sharing a common letter in the same row are significantly different (p<0.05). sol. stands for solvent-extraction; exp. stands for expeller-pressed.

Table 2 Mean particle sizes and bulk densities of six protein supplements ground through three sieves with different aperture sizes

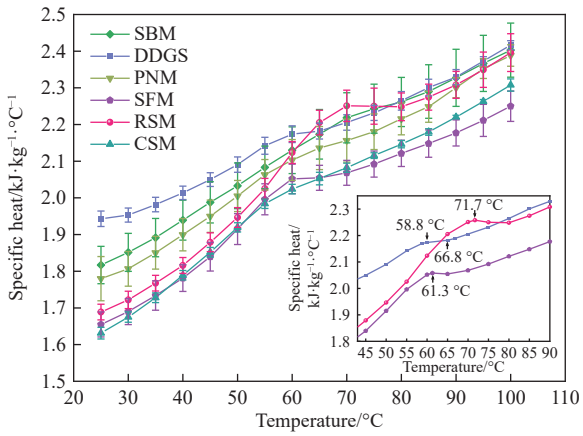
Protein supplement		<1.5 mm aperture size	<2.0 mm aperture size	<2.5 mm aperture size
Mean particle sizes/μm	SBM	370.62±0.10^{Ac}	412.93±2.86^{Ab}	483.70±1.48^{Aa}
	DDGS	335.55±4.35^{Bc}	358.84±2.03^{Cb}	370.34±1.30^{Ca}
	PNM	301.23±2.91^{Cc}	362.42±1.00^{Bb}	414.70±1.30^{Ba}
	SFM	260.97±0.47^{Dc}	295.60±0.78^{Db}	310.38±0.43^{Da}
	RSM	187.38±0.75^{Fc}	191.67±0.42^{Fb}	204.37±0.25^{Ea}
	CSM	248.37±0.92^{Ec}	266.96±1.04^{Eb}	307.97±3.51^{Da}
Bulk density/ g·cm^{-3}	SBM	0.720±0.001^{b}	0.720±0.002^{b}	0.720±0.003^{b}
	DDGS	0.643±0.003^{e}	0.643±0.002^{d}	0.643±0.003^{d}
	PNM	0.727±0.001^{a}	0.728±0.001^{a}	0.728±0.001^{a}
	SFM	0.556±0.003^{f}	0.557±0.002^{e}	0.557±0.004^{e}
	RSM	0.688±0.004^{d}	0.692±0.006^{c}	0.690±0.005^{c}
	CSM	0.692±0.003^{c}	0.691±0.004^{c}	0.692±0.004^{c}

Note: Values not sharing a common capital/lowercase letter in the same column/row are significantly different (p<0.05).

3.2 Specific heat (C_p)

3.2.1 Effect of temperature on C_p

The temperature-dependent C_p curves of six protein supplements through 2.0-mm sieve are shown in Figure 1. Obviously, the C_p of six feed meals increased with the increase in temperature. As the temperature raised from 25°C to 100°C, the C_p of SBM, DDGS and PNM increased from (1.817±0.051) to (2.403±0.074), (1.943±0.021) to (2.416±0.012), and (1.780±0.060) to (2.388±0.029) kJ/(kg·°C), respectively; and the C_p of SFM, RSM, and CSM grew from (1.655±0.034) to (2.250±0.041), (1.689±0.021) to (2.396±0.052), and (1.631±0.016) to (2.308±0.017) kJ/(kg·°C), respectively. The above C_p values fall into the range reported for many agricultural materials with low moisture content, such as cereal flours[13], red lentil seeds[18], stored canola seeds[15], energy feedstuffs[31], and cumin seeds[32], while the values are far lower than those of materials with high moisture content such as fruit pulps[14], white radish[23] and leafy vegetables[24]. For every 1°C increase in temperature, the C_p of SBM, DDGS, PNM, SFM, RSM, and CSM increased about 0.008, 0.006, 0.008, 0.008, 0.009, and 0.009 kJ/(kg·°C), respectively.



Note: SBM: Soybean meal; DDGS: Distillers dried grains with solubles; PNM: Peanut meal; SFM: Sunflower meal; RSM: Rapeseed meal; CSM: Cottonseed meal. Same below.

Figure 1 Specific heat of 6 protein supplements (<2.0 mm aperture size) as functions of temperature

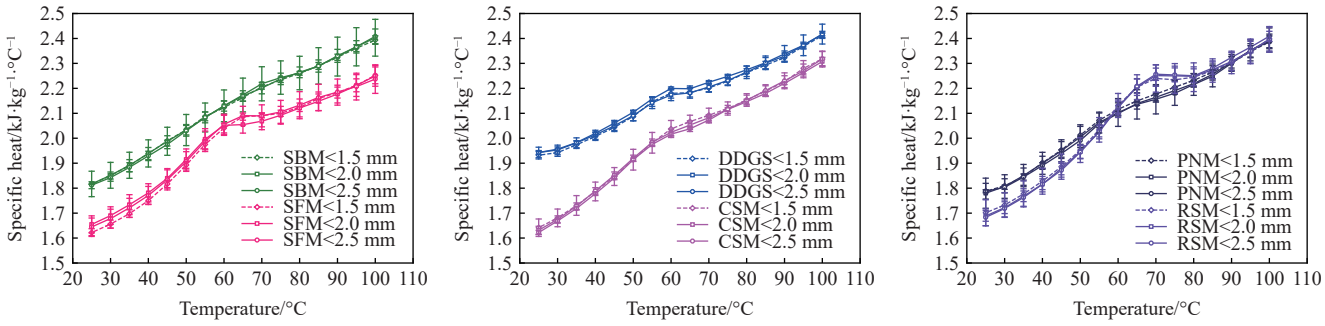
Distinguished from the other four feedstuffs, the C_p of SFM and RSM showed visible peaks at 61.3°C and 71.7°C, respectively. The corresponding values of C_p were 2.058 and 2.258 kJ/(kg·°C). Moreover, an inflection point at 58.8°C was observed on the C_p curve of DDGS, and there was a slow increase in C_p as temperature went up to 66.8°C. These phenomena may be closely related to the thermal denaturation of chemical components of the protein supplements.

The analysis of variance showed that the C_p of DDGS was significantly higher than that of the others at a temperature range of 25°C-40°C, while the C_p of SBM and PNM were greatly higher than that of SFM, RSM, and CSM. The C_p of RSM increased rapidly with increasing temperature from 65°C to 100°C, which was

significantly higher than that of SFM and CSM. Within the high-temperature range of 90°C-100°C, the C_p of SFM and CSM were significantly lower than that of the other four feedstuffs. However, no significant difference was observed among the C_p of RSM, SBM, DDGS, and PNM in the temperature range of 75°C-100°C.

3.2.2 Effect of particle size on C_p

The C_p curves of protein supplements under different particle sizes are shown in Figure 2. It is evident that the C_p of a certain protein supplement with different particle sizes shows an extremely similar variation with temperature. The analysis of variance revealed that there was no significant difference in C_p among different particle sizes at a given temperature for all the protein supplements (p>0.05). The results indicated that particle size had no significant effect on C_p for all the protein supplements.



a. SBM and SFM b. DDGS and CSM c. PNM and RSM

Figure 2 Specific heat curves of the protein supplements with three particle sizes

3.2.3 Models for C_p

Based on all the data of three particle sizes, regression models were developed to predict the C_p of protein supplements as functions of temperature as listed in Table 3. In consideration of the emergence of C_p peak, the C_p of SFM and RSM were modeled by piecewise non-linear functions to improve the prediction accuracy. The fitting models, with high R^2 values (R^2≥0.980) and low e (e≤ 0.659%) and RMSE values (RMSE≤0.015 kJ/(kg·°C)), fully reflected the relationships between the response C_p and the factor T (temperature) for all the protein supplements. As evident from Table 3, the C_p of DDGS increases linearly with the rise of temperature, which is in accordance with findings of previous research on cereal flours[13,31], canola with high oil content[20], *Jatropha curcas* L. kernels[22], white radish[23], shea nut kernel[36], pistachio nuts[37] and mushrooms[38]. It is also observed that the variations of C_p with temperature exhibit second-order polynomial relationships for PNM and SFM, and cubic polynomial relationships for SBM, RSM, and CSM. Similarly, Yu et al.[15] and Kong et al.[31] found second-order polynomial relationships between C_p and temperature for stored canola seeds (40°C-90°C) and beet pulp (25°C-100°C), respectively. The C_p following a quadratic polynomial relationship with temperature was also observed by Shrestha and Baik[21] for *Saponaria vaccaria* seed, by Yang et al.[39] for borage seeds, and by Singh and Goswami[32] for cumin seed at low moisture content levels (1.8%-11.1% d.b.). Kong et al.[31] established cubic polynomial models for C_p prediction of wheat bran, rice bran, and cassava residue with temperature ranging from 25°C to 100°C.

Table 3 Regression equations and statistical information for the specific heat of six protein supplements

Protein supplements	Regression model	F value	Degree of freedom	RMSE/ kJ·(kg⁻¹·°C⁻¹)	e/ %	R^2
SBM	$C_p = 1.55674 + 0.00995T - 1.60776\times10^{-7}T^3$	8808.413	47	0.009	0.366	0.997
DDGS	$C_p = 1.77333 + 0.00631T$	4106.324	47	0.015	0.563	0.989
PNM	$C_p = 1.50394 + 0.01112T - 2.40745\times10^{-5}T^2$	3337.217	47	0.015	0.659	0.993
SFM	$C_p = 1.54721 + 0.00014T^2$ (25°C-60°C)	2523.876	23	0.013	0.597	0.991
	$C_p = 2.10389 - 0.00422T + 5.64008\times10^{-5}T^2$ (60°C-100°C)	586.171	26	0.009	0.329	0.980
RSM	$C_p = 1.98911 - 0.02884T + 0.00081T^2 - 4.86959\times10^{-6}T^3$ (25°C-70°C)	2933.161	29	0.010	0.448	0.997
	$C_p = 2.90810 - 0.01371T + 8.67713\times10^{-7}T^3$ (70°C-100°C)	440.924	20	0.008	0.289	0.980
CSM	$C_p = 1.17388 + 0.02095T - 0.00015T^2 + 5.62149\times10^{-7}T^3$	2979.139	47	0.015	0.639	0.995

Note: C_p means specific heat, kJ/(kg·°C); T means temperature, °C; RMSE is root mean square error; e represents mean relative percent error; R^2 is coefficient of determination.

3.2.4 Heat absorption

The C_p of protein supplements is directly associated with the additional amount of steam required to heat up per ton of feed to the desired temperature during the conditioning process. Currently, the common conditioning temperature used in the production of pelleted feeds for Livestock ranges from 65°C to 85°C. Therefore, theoretical quantities of heat absorption of six protein feedstuffs during temperature rise were calculated by integrating the developed functions of C_p (Table 3) in corresponding temperature ranges and the results are shown in Figure 3.

It can be observed that the heat needed for temperature rising from 25°C to 75°C and 85°C was about 1.28 times and 1.57 times as much as that from 25°C to 65°C, respectively. At a temperature-rise range of 25°C-75°C, the quantity of heat absorption required for DDGS was the highest (104 441.500 kJ/t), followed by SBM (101 455.938 kJ/t), while that for SFM was the lowest (94 659.288 kJ/t). To rise to the same temperature, DDGS needed to absorb 3% more heat than that required for SBM, while PNM, SFM, RSM, and CSM needed just 98%, 93%, 97%, and 94% of the heat that for SBM, respectively. Thus, when partly replacing SBM with other

protein supplements or changing the conditioning temperature, it is necessary to quantificationally adjust the additional amount of steam according to the aforementioned research findings to ensure that the expectant conditioning effect can be achieved quickly and accurately.

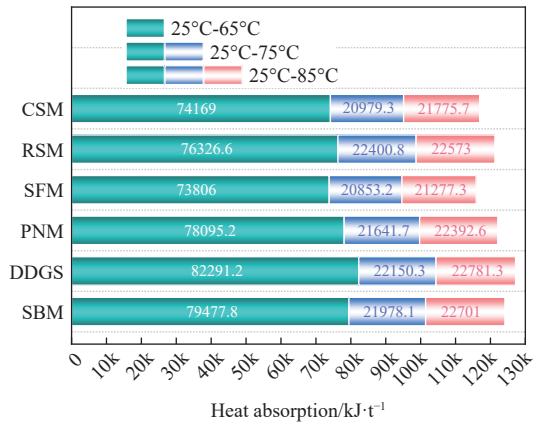


Figure 3 Theoretical quantities of heat absorption of 6 protein supplements during temperature rise from 25°C to 65°C/75°C/85°C

3.3 Thermal conductivity (k_b)

3.3.1 Effect of temperature on k_b

The variations of k_b with temperature for six feed meals through a 2.0-mm sieve are shown in Figure 4. It is obvious that the k_b of all feedstuffs exhibit positive non-linear correlations with temperature in the range of 25°C-100°C as reported by other research workers on some agricultural products[15,21,32]. This phenomenon could be attributed to the fact that ions and dipoles in materials displayed higher lattice vibrations at higher temperatures accelerating heat transfer[15]. At high temperature of 55°C-100°C, the sharp increase of k_b might stem from the occurrence of protein denaturation and nonenzymatic browning in protein supplements, which could result in the increase of stickiness, swelling of particles, and decrease of porosity.

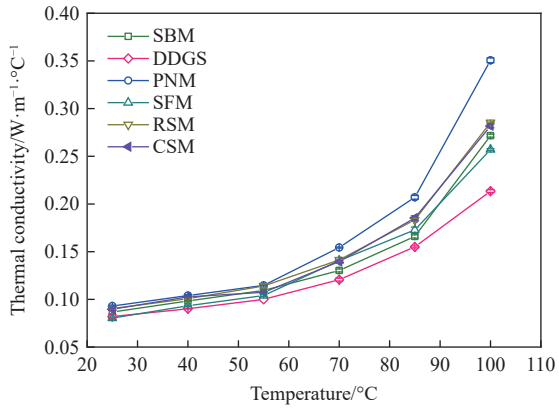


Figure 4 Thermal conductivities of 6 protein supplements (<2.0 mm aperture size) as functions of temperature

As displayed in Figure 4 the k_b rose with increasing temperature from 0.087 to 0.272 W/(m·°C) for SBM, 0.082 to 0.214 W/(m·°C) for DDGS, 0.093 to 0.351 W/(m·°C) for PNM, 0.080 to 0.257 W/(m·°C) for SFM, 0.090 to 0.285 W/(m·°C) for RSM and 0.090 to 0.282 W/(m·°C) for CSM. The analysis of variance showed that the k_b of PNM was significantly higher than that of the other feedstuffs in high-temperature range of 70°C-100°C, while the k_b of DDGS was significantly lower than that of the others (p<0.05). This result was ascribed to the fact that particulate materials in compact form transfer heat better than in loose form due to fewer air voids in the matrix[15,21]. Compared to the other feedstuffs, PNM had a higher stickiness at high temperatures and a significantly higher bulk density (Table 2), which may result in a considerably higher k_b. The lower k_b of DDGS was the consequence of a lower bulk density and a greater particle size, which are unfavorable for heat transfer between particles.

3.3.2 Effect of particle size on k_b

Figure 5 shows the differences in k_b of six feedstuffs among different particle sizes with the same bulk density at 25°C, 55°C, and 85°C. The k_b demonstrated significant differences among three particle sizes for PNM and CSM at 25°C; for DDGS, PNM, and SFM at 55°C; for SBM, DDGS, PNM, and SFM at 85°C (p<0.05). It can be found in Figure 5 that the k_b value tends to decrease gradually with increase in particle size for the same feedstuff, especially at high temperatures. This may be due to the fact that the finer feed meal is more prone to protein denaturation at high temperature compared to the coarse one, which makes the meal structure stickier and more compact, resulting in a higher value of k_b.

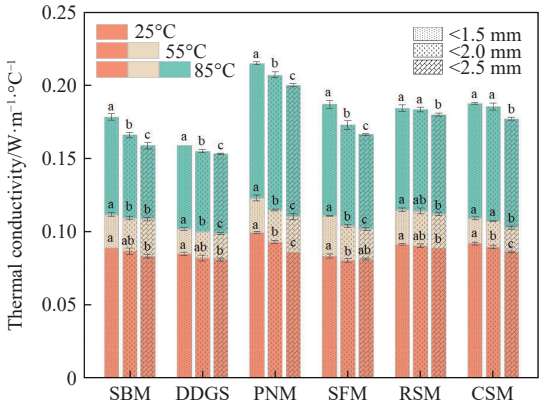


Figure 5 Differences in thermal conductivities of 6 protein supplements among three particle sizes at 25°C, 55°C, and 85°C

3.3.3 Models for k_b

The relationships between k_b and temperature were modeled for six protein supplements (<2.0 mm aperture size) as given in Table 4. These models tend to give predictions that are parallel to the experimental data based on the observation of high values of R^2 (0.994-0.998) as well as low values of RMSE (0.003-0.005 W/(m·°C)) and e (1.964%-3.456%). It's worth noting that the variations of k_b with increasing temperature from 25°C to 100°C follow cubic polynomial relationships for all the feedstuffs, which are consistent with the results reported by Yu et al.[15] for stored canola seeds (40°C-90°C), and by Kong et al.[31] for four cereal grains and beet pulp (25°C-100°C).

The quadratic and linear relationships between k_b and temperature had been reported for other agricultural materials. The research on cumin seeds showed a quadratic relationship between k_b and temperature from −50°C to 50°C[32]. Shrestha and Baik[21] developed a quadratic polynomial equation to measure the k_b of *Saponaria vaccaria* seeds as a function of temperature in the range of 25°C-55°C. A linear increase in k_b with rising temperature was reported by Jian et al.[40] for canola seeds with high oil content in storage temperature range (−10°C-30°C). Furthermore, the k_b of white radishes (*R. raphanistrum*) and mushrooms (*Pleurotus florida*) also followed positive linear relationships with temperature in the corresponding range (20°C-80°C; 40°C-70°C, respectively) according to studies by Obot et al.[23] and Shrivastava and Datta[38], respectively.

Table 4 Regression equations and statistical information for temperature-dependent k_b and α of six protein supplements (<2.0 mm aperture size)

Protein supplements	Regression model	*F* value	Degree of freedom	RMSE	*e*/%	R^2
SBM	$k_b = 0.00604T - 0.00012T^2 + 9.10793\times10^{-7}T^3$	1612.941	6	0.004	2.308	0.996
	$\alpha = 1.31123 + 0.37392T - 0.00793T^2 + 5.61893\times10^{-5}T^3$	244.918	5	0.164	1.513	0.997
DDGS	$k_b = 0.07862 + 1.31745\times10^{-7}T^3$	952.350	5	0.003	2.061	0.996
	$\alpha = 6.78566 - 0.00045T^2 + 1.13462\times10^{-5}T^3$	1042.973	5	0.094	1.064	0.999
PNM	$k_b = 0.00652T - 0.00014T^2 + 1.10516\times10^{-6}T^3$	1419.405	6	0.005	2.414	0.997
	$\alpha = 0.47789T - 0.00999T^2 + 7.23012\times10^{-5}T^3$	1150.215	6	0.346	3.476	0.994
SFM	$k_b = 0.00484T - 8.72484\times10^{-5}T^2 + 6.44915\times10^{-7}T^3$	1028.717	6	0.005	3.456	0.994
	$\alpha = 9.20531 - 0.00098T^2 + 2.10939\times10^{-5}T^3$	150.209	5	0.415	3.392	0.990
RSM	$k_b = 0.00590T - 0.00012T^2 + 8.62678\times10^{-7}T^3$	3078.111	6	0.003	1.964	0.998
	$\alpha = 4.02420 + 0.27504T - 0.00640T^2 + 4.98820T^3$	6385.354	5	0.035	0.339	1.000
CSM	$k_b = 0.00587T - 0.00012T^2 + 8.63569\times10^{-7}T^3$	2213.128	6	0.004	2.195	0.997
	$\alpha = 8.82908 - 0.00157T^2 + 2.44039\times10^{-5}T^3$	271.860	5	0.257	2.594	0.995

Note: k_b means thermal conductivity, W/(m·°C); α means thermal diffusivity, 10^{-8}·m²/s; The unit of RMSE is the same as k_b or α.

The results of variance analysis (Table A2) for k_b show that temperature and particle size as well as the interaction term significantly affect the k_b of protein feedstuffs at 0.1% level. Based on temperature-dependent models (Table 4), empirical equations considering the combined influence of particle size and temperature were developed to describe k_b of all the feedstuffs (Table 5). The regression models, with low e (e≤2.619%) and RMSE values (RMSE≤0.005 W/(m·°C)) as well as high R^2 values (R^2≥0.995), precisely predicted k_b of all the feedstuffs within the ranges of temperature and particle size involved in this study. It is observed that, for a given temperature, the k_b displays linear relationships with particle size for DDGS, PNM, and CSM, and quadratic polynomial relationships for the others.

3.4 Thermal diffusivity (α)

3.4.1 Effect of temperature on α

The α of six feed meals through a 2.0-mm sieve were plotted against temperature as shown in Figure 6. At the temperature range of 25°C-100°C, the α values ranged from 6.637×10^{-8} to 15.706×10^{-8} m²/s for SBM, 6.578×10^{-8} to 13.691×10^{-8} m²/s for DDGS and 7.184×10^{-8} to 20.136×10^{-8} m²/s for PNM; while the α values of SFM, RSM, and CSM varied in the range of 8.698×10^{-8}-20.643×10^{-8}, 7.694×10^{-8}-17.417×10^{-8}, and 8.030×10^{-8}-17.574×10^{-8} m²/s, respectively. The α increased slowly with temperature rise in the range of 25°C-55°C, while increased sharply in the range of 55°C-100°C for all the feedstuffs. It should, however, be noted that the α of CSM presented a slight decline from 8.290×10^{-8} to

Table 5 Regression equations and statistical information for k_b and α of six protein supplements as functions of temperature and particle size

Protein supplements	Regression model	Degree of freedom	RMSE	*e*/%	R^2
SBM	$k_b = 0.079\,90 + 0.007\,81T - 0.000\,14T^2 + 1.002\,29\times10^{-6}T^3 - 0.000\,49d_{gw} + 5.871\,37\times10^{-7}{d_{gw}}^2 - 1.651\,96\times10^{-6}Td_{gw}$	17	0.004	2.619	0.996
	$\alpha = 8.488\,12 + 0.408\,04T - 0.007\,89T^2 + 5.619\,57\times10^{-5}T^3 - 0.033\,75d_{gw} + 3.930\,22\times10^{-5}{d_{gw}}^2 - 8.781\,87\times10^{-5}Td_{gw}$	17	0.181	1.842	0.997
DDGS	$k_b = 0.127\,36 + 1.509\,05\times10^{-7}T^3 - 0.000\,11d_{gw} - 7.458\,94\times10^{-7}Td_{gw}$	17	0.002	1.658	0.998
	$\alpha = 22.898\,88 - 0.000\,29T^2 + 1.065\,57\times10^{-5}T^3 - 0.079\,66d_{gw} + 9.767\,94\times10^{-5}{d_{gw}}^2 - 2.558\,24\times10^{-5}Td_{gw}$	17	0.132	1.386	0.997
PNM	$k_b = 0.012\,85 + 0.007\,49T - 0.000\,15T^2 + 1.164\,15\times10^{-6}T^3 - 6.870\,55\times10^{-5}d_{gw} - 7.577\,88\times10^{-7}Td_{gw}$	17	0.005	2.573	0.997
	$\alpha = 4.969\,28 + 0.332\,98T - 0.007\,46T^2 + 5.942\,30\times10^{-5}T^3 - 0.006\,28d_{gw} - 2.101\,14\times10^{-5}Td_{gw}$	17	0.276	2.216	0.996
SFM	$k_b = 0.085\,96 + 0.004\,67T - 5.798\,55\times10^{-5}T^2 + 5.011\,43\times10^{-7}T^3 - 0.000\,52d_{gw} + 1.154\,52\times10^{-6}{d_{gw}}^2 - 5.596\,43\times10^{-6}Td_{gw}$	17	0.004	2.330	0.995
	$\alpha = 24.247\,71 - 0.000\,41T^2 + 1.896\,22\times10^{-5}T^3 - 0.088\,89d_{gw} + 0.000\,14{d_{gw}}^2 - 0.000\,15Td_{gw}$	17	0.427	3.144	0.990
RSM	$k_b = 0.015\,68 + 0.006\,07T - 0.000\,10T^2 + 7.764\,06\times10^{-7}T^3 - 0.000\,12d_{gw} + 5.995\,41\times10^{-7}{d_{gw}}^2 - 5.434\,65\times10^{-6}Td_{gw}$	17	0.003	1.620	0.998
	$\alpha = 18.576\,78 + 0.339\,59T - 0.006\,02T^2 + 4.725\,29\times10^{-5}T^3 - 0.155\,28d_{gw} + 0.000\,42{d_{gw}}^2 - 0.000\,42Td_{gw}$	17	0.074	0.563	1.000
CSM	$k_b = 0.046\,86 + 0.004\,24T - 8.471\,88\times10^{-5}T^2 + 6.977\,59\times10^{-7}T^3 - 5.322\,74\times10^{-5}d_{gw} - 9.805\,47\times10^{-7}Td_{gw}$	17	0.003	2.017	0.998
	$\alpha = 11.215\,44 - 0.001\,71T^2 + 2.507\,37\times10^{-5}T^3 - 0.009\,28d_{gw} + 2.865\,33\times10^{-5}Td_{gw}$	17	0.295	2.934	0.993

Note: d_{gw} means particle size, μm.

7.810×10^{-8} m²/s in the range of 40°C-55°C. The increase of α could be explained by the fact that the growth rates of k_b with temperature were higher than that of C_p[15]. Shrestha and Baik[21] reported the dominant contribution of k_b variation to the α of *Saponaria vaccaria* seed particles.

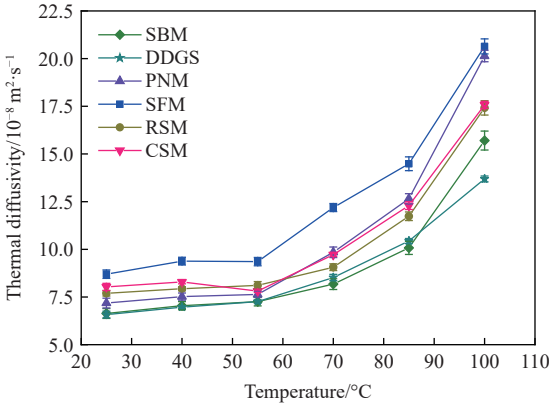
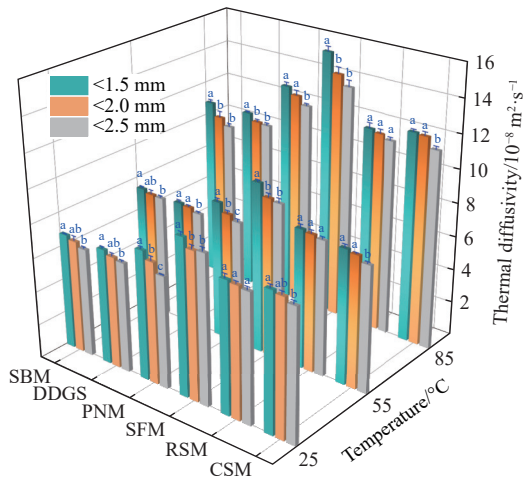


Figure 6 Thermal diffusivities of 6 protein supplements (<2.0 mm aperture size) as functions of temperature

The product of C_p and ρ_b is called volumetric heat capacity which symbolizes the ability of a material to store heat. Therefore, the α of a material reflects the ratio of heat conduct capability to heat storage capability. The α of SFM was noticeably higher than those of the other feedstuffs at the studied temperature range as a consequence of the observed low values of C_p and ρ_b, which indicated that SFM had a relatively weaker capacity for heat storage. The high values of C_p and low values of k_b contributed to the lower α of SBM and DDGS compared to the other four feedstuffs. Furthermore, the greater ρ_b of SBM was another main factor resulting in the lower α. These results suggest that it is not easy for SBM to heat up or cool down owing to a bad capacity for conducting heat, but a strong capacity for storing heat.

3.4.2 Effect of particle size on α

Figure 7 compares the differences in α of six feedstuffs among different particle sizes at 25°C, 55°C, and 85°C. The α exhibited descending trends with increasing particle size for all the feedstuffs, especially for PNM, which appeared significant decline in α at 25°C (7.637×10^{-8}–6.605×10^{-8} m²/s) and 55°C (8.159×10^{-8}–7.350×10^{-8} m²/s) ($p<0.05$). There was no significant difference in α of RSM among the three particle sizes at all three temperatures.



Note: The three adjacent bars not sharing a common letter represent significant differences in α at different particle sizes for the same protein supplement ($p<0.05$).

Figure 7 Differences in thermal diffusivities of 6 protein supplements among three particle sizes at 25°C, 55°C, and 85°C

3.4.3 Models for α

Regression equations related to temperature were developed for the α of six feedstuffs (<2.0 mm aperture size) and statistical information is summarized in Table 4. The models could be used to reasonably predict the α of the feedstuffs since the high values of R^2 (0.990-1.000) and low values of e (0.339%-3.392%) and RMSE (0.035×10^{-8}-0.415×10^{-8} m²/s) were observed. It is apparent that α of all the feedstuffs show cubic polynomial relationships with temperature (25°C-100°C). This finding is qualitatively similar to that obtained by Yu et al.[15] and Kong et al.[31] who also developed, respectively, cubic polynomial equations for describing changes in α of canola seeds and energy feedstuffs (four cereal grains, cassava residue, and beet pulp) over temperature. In addition, quadratic polynomial relationships between α and temperature were reported by Shrestha and Baik[21] for *Saponaria vaccaria* seed in the range of 25°C-55°C and by Singh and Goswami[32] for cumin seed in the range of −50°C-50°C. Obot et al.[23] found that the α of white radish increased linearly with temperature in the range of 20°C-80°C.

Likewise, empirical equations were established to describe the combined effect of particle size and temperature on the α of the protein supplements as given in Table 5. The statistical information of high R^2 values (0.990-1.000) and low e values (0.563%-3.144%) suggested that developed models could be directly used to predict α of the feedstuffs with sufficient accuracy.

4 Conclusions

In this study, differences in the thermophysical properties among six protein supplements (SBM, DDGS, PNM, SFM, RSM, and CSM) were investigated and the influences of temperature and particle size on these properties were evaluated. The C_p of all the feedstuffs increased with increasing temperature (25°C-100°C) and ranged from 1.622 to 2.417 kJ/(kg·°C). Particle size had no significant effect on C_p for all the feedstuffs, thus C_p was only modeled as a function of temperature. To rise to the same temperature, DDGS needed to absorb 3% more heat than that required for SBM. However, the rest four feedstuffs just needed to absorb 93%-98% of the heat for SBM. It was not easy for SBM to heat up or cool down in comparison with other protein supplements. The k_b and α of all the feedstuffs generally exhibited positive non-linear correlations with temperature and varied from 0.080 to 0.362 W/(m·°C) and 6.379×10^{-8} to 21.984×10^{-8} m²/s, respectively. Descending trends in k_b and α were observed with increasing particle size for a certain feedstuff at the same bulk density. Empirical equations with only statistically significant terms were developed to measure the combined effect of temperature and particle size on k_b and α. These proposed models adequately fitted the experimental data with mean relative percent errors between 0.289% and 0.659% for C_p, 1.620% and 2.619% for k_b, and 0.563% and 3.144% for α, which can provide accurate data for heat transfer calculations and simulations in the thermal processing of pelleted feeds.

The study of the thermal properties of protein feedstuffs will facilitate the equipment design, parameter optimization, and simulation for thermal processing involved in the manufacture of pelleted feeds like conditioning and cooling, especially when energy consumption and pellet quality are the main consideration. However, considering the limited category and source of protein supplements in this work, further study should be taken on a wider variety of protein supplements from different manufacturers.

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Appendix

Table A1 Experimental moisture contents of the feed meals after drying or moistening treatment (% wet basis)

Protein supplements	Different particle sizes		
	<1.5 mm aperture size	<2.0 mm aperture size	<2.5 mm aperture size
SBM	11.01±0.02	11.10±0.01	11.06±0.01
DDG	11.04±0.13	11.04±0.08	11.09±0.08
PNM	11.03±0.00	11.10±0.01	11.04±0.03
SFM	11.01±0.03	11.10±0.10	11.04±0.06
RSM	11.03±0.03	11.06±0.08	11.03±0.03
CSM	11.04±0.03	11.04±0.01	11.11±0.02

Note: <1.5/2.0/2.5 mm aperture size means the protein supplements are being ground through sieves with aperture sizes of 1.5/2.0/2.5 mm.

Table A2 Analysis of variance for effects of temperature and particle size on thermal conductivities of the protein supplements

Source of variation	Degrees of freedom	Sum of square	Mean square	*F* value	*p* value
Intercept	1	7.098	7.098	3 462 048.122***	0
Temperature (T)	5	1.378	0.276	134 373.552***	0
Particle size (d_{gw})	17	0.059	0.003	1684.387***	0
$T\times d_{gw}$	85	0.058	0.001	330.975***	0
Error	216	4.429×10^{-4}	2.050×10^{-6}		
Total	324	8.593			

Note: *** Significant at 0.1% level. $T\times d_{gw}$ represents the interaction between temperature and particle size.