

MEASUREMENT AND MODEL VALIDATION OF SPECIFIC HEAT OF XANTHAN GUM USING JOULES CALORIMETER METHOD

K. Muthamizhi¹, P. Kalaichelvi², A. Arunagiri³, A. Rudhra⁴

¹ Researchscholar, ^{2,3} Associate Professor, ⁴ B. Tech, Chemical Engg Dept, National Institute of Technology, Tamil Nadu, India, tamilkrypal@gmail.com, kalai@nitt.edu, aagiri@nitt.edu, rudhra.nitt@gmail.com.

Abstract

The purpose of this paper is to examine the concentration and temperature influence on the specific heat of xanthan gum and to propose a model equation for estimating the specific heat of xanthan gum at different concentration and temperature using the Joules calorimeter method. Joules calorimeter method was tested with distilled water for accuracy and reliability before applying to xanthan gum samples which varied in concentration. The specific heat of xanthan gum needs to be known for evaluating the design and modeling aspects of heat transfer processes of refrigeration, freezing, heating, pasteurization and drying. The specific heat of xanthan gum increases with an increase in temperature (293.15 - 333.15K) and concentration (0.1 - 0.6 %w/w). The minimum value of specific heat of xanthan gum was 4.133 KJ/kg K at 300.25K with a concentration of 0.2 %w/w, whereas the maximum value of specific heat of xanthan gum was 7.459 KJ/kg K at 333.95K with a concentration of 0.5 %w/w. The specific heat capacity of xanthan gum is compared with that of pure water at 308.15, 318.15 and 328.15 K and literature available model at 293.15 - 333.15 K for 0.1 - 0.6 %w/w concentration of xanthan gum. The influence of operating parameters on the specific heat of xanthan gum was determined by employing a central composite rotatable design in response surface methodology (CCRD-RSM). The new model equation obtained for estimating the specific heat using RSM possesses good agreement with experimental data with a regression coefficient of 0.9774.

Index Terms: Specific heat, Xanthan gum, Pseudo plastic fluid and Response surface methodology etc....

1. INTRODUCTION

Xanthan gum is a high molecular weight polysaccharide, produced by the bacterium *Xanthomonas campestris* [1-3] and it can be used to add texture to food [4]. Texture is one of the most vital properties to characterize the quality of food products.

Xanthan gum is soluble in cold and hot water and the powder of xanthan gum has a strong tendency to form lumps when added with water.

This specific property of the gum needs intensive agitation in order to avoid the formation of lumps like hydrocolloids [5]. The xanthan gum primary structure is a linear β -(1-4) -D-glucose, which is the backbone of xanthan gum and it has similar characteristics as cellulose [6-7]. Xanthan gum forms a twin stranded, right-handed fivefold helix and the stability of the helix is strongly affected by the Ionic environment [8].

Xanthan gum is used in food, chemicals, petrochemicals, polymers, cosmetic and pharmaceutical industry, for a number of key reasons, including emulsion stabilization, temperature stability, and compatibility with food ingredients [5-9].

Xanthan gum has pseudo-plastic fluid property - both two- and three-dimensional behavior - which is different from the

behavior of a Newtonian fluid. The particle of pseudo-plastic fluid contains dissolved or dispersed particles, which have random orientations in the fluid at rest, but get lined up when the fluid is sheared. This property allows it to pour, pump and facilitates ease of swallowing [10].

The heating and cooling of foods are the most important unit operations in food processing industries [11-15]. Due to the need for the information about the thermo-physical properties of foods, the design of food products has been limited [12&16]. Specific heat, thermal conductivity, density, viscosity, surface tension and coefficient of thermal expansion are the most important thermo-physical properties of pseudo-plastic fluids. As of now, the limited value of thermo-physical measurement has been conducted for dilute and aqueous solutions of moderate concentration of typically used polymers such as polyethylene oxide, carbopol, polyacrylamide, and carboxymethyl cellulose [17]. The specific heat of the food products strongly depends upon their temperature and concentration [18-21].

Total heat required to raise 1 g of substance temperature by 1° C is called specific heat [22]. Specific heat is the most important thermodynamic parameter in heat transfer calculations and it increases when the temperature increases. It is case sensitive to the composition of the ingredients (if the specific heat of individual components is different) and to the

interactions between the components of the material [14]. Similarly, for various foods and biological materials, specific heat increases with increase in temperature [23-27]. The specific heat capacity of most of the substances depends on the state variable and particularly it is dependent on temperature [28]. Semmar *et al.*, (2004) determined the specific heat of CMC (Carboxymethyl cellulose) and CPE (carboxy-poly-ethylene) solution as a function of temperature and concentration [29].

The response surface methodology is a statistical and mathematical technique used for modeling and optimization of processes in which a response of interest is influenced by several variables. The primary function of RSM is to postulate a suitable approximation for the true functional relationship between the response and the set of independent variables. The two most important designs extensively used in Response surface methodology are Central Composite Design (CCD) and Box–Behnken Design (BBD) [30].

The foremost significant part to design any process involving pseudo-plastic fluids on an industrial scale is thermo-physical properties. In the present study, the specific heat capacities of xanthan gum solutions were measured, in the temperature range of 293.15 K - 333.15 K and concentration in the range of 0.1-0.6 %w/w compared with water. A more general relationship that takes into account, the temperature and the concentration has been established with an average relative error of 0.130% using RSM-CCD.

2. MATERIALS AND METHODS

2.1 Materials and Solution Preparation

Xanthan gum pure (food grade) was purchased from LOBA Chemie. Xanthan gum (1 kg) had a particle size (100-200 mesh), ash (6.5-16%), pH of 1% solution in water (6-8), nitrogen (1.5%), pyruvic acid (2.40%) and heavy metals, (0.001%).

A known amount of xanthan gum was weighed (CyberlabTM) and the weighed sample was dissolved in 350 ml distilled water to obtain different concentrations ranging from 0.1-0.6 % w/w. The mixture was stirred well for 1 hour in a magnetic stirrer at room temperature until the solute was dissolved completely. The xanthan gum mixture was transferred into copper calorimeter and the measurements of specific heat were done.

Joules calorimeter was supplied by *Precision scientific & Co*, Trichy. Joules calorimeter is electrically heated calorimeter, such as that used in the Griffiths method and it consists of a copper vessel of 7.62 X 5.08 cm with the outer vessel as a superior quality teak wood box of size 9.5 X 9.5 X 11.5 cm fitted to the Bakelite lid with a hole for the thermometer and stirrer.

2.2 Experimental Procedure

A Joule's calorimeter apparatus was used; which works on adiabatic principle [22]. It was calibrated using distilled water as a sample fluid to test the accuracy and reliability of the process. A schematic diagram of the Joules Calorimeter is shown in Fig. 1.

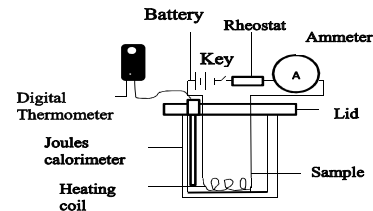


Fig -1: Schematic diagram of a Joules calorimeter

Experimentally, the amount of electrical joule heat generated in a circuit element of resistance R is measured by calorimetric methods. After measuring the mass of the empty calorimeter, the inner calorimeter cup was filled with 350 ml of xanthan gum.

The calorimeter was placed in a wooden box. The temperature probe was suspended to ensure that it does not touch the coil or interfere with the stirrer. The battery was connected in series with joules calorimeter, a rheostat, ammeter and plug key. The switch was turned on for the power supply, voltage, current and the time taken for equal intervals of temperature rise were measured and the specific heat was calculated by using equation (1) where C_p , M , w , E , I , t , T_f , T_i and C_{p_m} are Specific heat of the fluid, Mass of the fluid, weight of the calorimeter, Voltage, Current, time, Final temperature and Initial temperature and specific heat of the material.

$$C_p = \frac{1}{M} \left(\frac{(E \times I \times t)}{(T_f - T_i)} - (w \times C_{p_m}) \right) \quad (1)$$

2.3 Experimental Design

To study the effect of operating variables on the specific heat of xanthan gum, Design Expert Software (8.0.6) was used. Independent variables, i.e. temperature and concentration were coded at five levels between -2 and +2, wherein the temperature (T) in the range of 293.15 K - 333.15 K and concentration in the range of 0.1 - 0.6 %w/w (C_x), respectively. The experimental range and independent process variable levels of xanthan gum solution are presented in Table 1.

A total number of 13 runs were obtained with two factors using the Design Expert Software. To evaluate the pure error, eight experiments were carried out with five replications at the design center in randomized order.

3. RESULTS AND DISCUSSIONS

The specific heat of any solution is influenced by temperature, concentration, interaction of pseudo-plastic fluid components and others.

The following sections 3.1 and 3.2 describe the influence of the concentration and temperature on the specific heat of xanthan gum solution.

Table -1: Range of experimental and independent process variable levels for xanthan gum solution

Factors	Unit	Levels and range				
		-2	-1	0	1	2
Temp	K	284.86	293.15	313.15	333.15	341.43
Conc	%	0	0.1	0.35	0.6	0.7

3.1 Effects of Temperature of Specific Heat of Xanthan Gum Solution

The temperature and concentration influence of the specific heat of xanthan gum solution was shown in Fig.2. It can be inferred from Fig. 2 that the specific heat of the xanthan gum solution increases when temperature increases for 0.1 % w/w, 0.3 % w/w and 0.5 % w/w. A similar trend was observed for 0.2 % w/w, 0.4 % w/w and 0.6 % w/w.

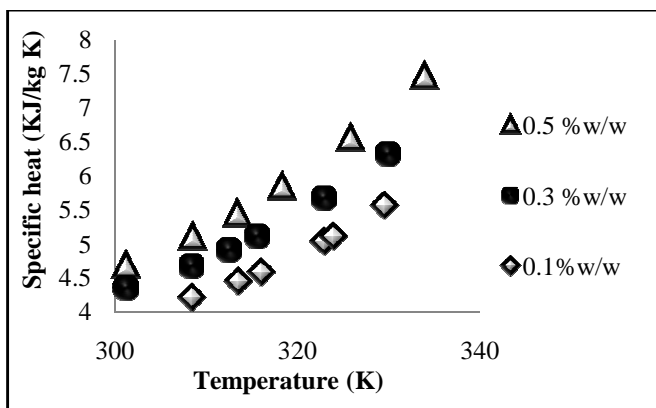


Fig -2: Temperature and concentration influence on the specific heat of xanthan gum solution

Specific heat depends on the structure of the xanthan gum. During heating, xanthan gum will expand, for the expansion, a little amount of heat also supplied to the work required; this will increase the specific heat of xanthan gum solution with a maximum value of 7.4595 KJ/kg K for 0.5 % w/w at 333.95 K. As the xanthan gum heats up, the average temperature of the xanthan gum molecules increases, so when they collide, they are more likely to impart enough energy to allow rotation and vibration to occur as the energy jumps to a higher state. Once the rotation is induced, it contributes to the internal

energy and raises the specific heat. This is typical for all mixtures higher the temperature higher the specific heat [31].

3.2 Effects of Concentration on Specific Heat of Xanthan Gum Solution

Fig. 2 shows that the specific heat of xanthan gum solution increases when the concentration increases for 0.1 % w/w, 0.3 % w/w and 0.5 % w/w at 293.15 K - 333.15 K. A similar trend was observed for 0.2 % w/w, 0.4 % w/w and 0.6 % w/w at 293.15 K - 333.15 K. It was observed from the Fig. 2 that the specific heat increases generally with an increase in xanthan gum concentration. When concentration increased more heat was required to raise the temperature of the xanthan gum by one degree Kelvin. Increase in specific heat due to the increase in concentration of xanthan gum increased the heat requirement of samples. Thus the sample with 0.6 % w/w xanthan gum required more heat to raise its temperature than the sample with 0.1 % w/w [32].

3.3 Comparison of Experimental Values of Specific Heat of Xanthan Gum Solution to the Specific Heat of Water

The specific heat of xanthan gum solution (0.1 % w/w, 0.3 % w/w and 0.5 % w/w) was compared with water at different temperatures (308.15 K, 318.15 K and 328.15 K) as depicted in Fig. 3.

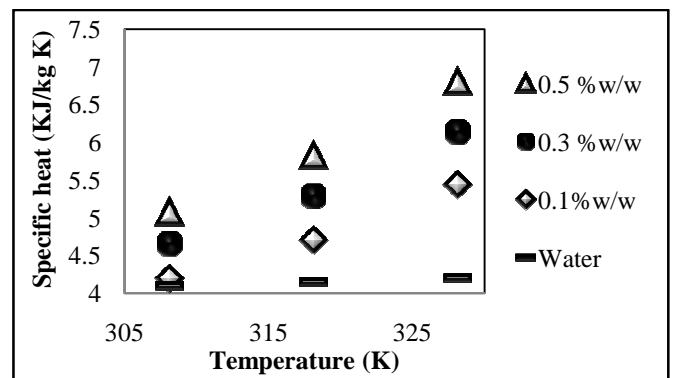


Fig -3: Specific heat of xanthan gum solution compared with water at different temperature (308.15 K, 318.15 K and 328.15K)

The specific heat of xanthan gum solution was higher compared to the specific heat of water at all concentrations considered in the present study. Bellet (1975) and Raynaud (1985) narrated that the specific heat values of solutions are higher than the specific heat of water [33-34].

Semmarel *et al.*, (2004) concluded that even for low values of polymeric solution mass concentration, their specific heat

cannot be regarded as equal to that of pure water [29]. Even if water has the highest specific heat of all liquids except ammonia, in the presence of water molecules, molecular chains of xanthan gum possess an energy potential which increases with temperature and concentration, thus it has a high specific heat compared to water.

3.4 Comparison Of Experimental Values Of Specific Heat Of Xanthan Gum Solution To The Reference Model Equation Of Semmar *et al.*, (2004)

The experimental data of specific heat of xanthan gum was also tested using second order polynomial model equation reported for CMC and CPE solution by Semmar *et al.*, (2004) [29].

Semmar *et al.*, (2004) established the following model equation where Cp, T, T₀, A & B, D and X are Specific heat, Temperature, Temperature of pure water (T₀ =273.15K), Coefficients of temperature, Coefficients of temperature, Coefficient of concentration and concentration [29].

$$Cp(T, X) = A(T - T_0)^2 + B(T - T_0) + Cp(T_0) \left[\frac{DX}{1+DX} \right] \quad (2)$$

- A = 0.015
- B = 5.45
- D = -0.013 for 18gl⁻¹

Table 2 shows the comparison of experimental results and specific heat of xanthan gum solution estimated using the model equation proposed by Semmar *et al.*, (2004). Comparison between experimental and predicted specific heat value of xanthan gum solution reveals that the model equation predicts the specific heat of xanthan gum solution in the database with an absolute average deviation of 14.1. Hence, response surface methodology was used to develop an empirical model equation for specific heat of xanthan gum solution as a function of temperature and concentration.

3.5 Development of a model equation for Specific heat of xanthan gum solution using RSM

The analysis was done using the Design Expert Software (8.0.6) which is focused on how the specific heats of xanthan gum solution are influenced by independent variables, i.e., temperature and concentration.

Analysis of Variance (ANOVA)

ANOVA was used to determine the significant effects of temperature and concentration and the results are presented in Table 3.

It shows the comparison between the predicted result and experimental result of specific heat of xanthan gum solution. It

can be observed from Table 4 that F-values in regression analysis are found to be higher for specific heat of xanthan gum solution.

Table -2: Comparison between the experimental result and specific heat of xanthan gum solution estimated using the model equation proposed by semmar *et al.*, (2004)

The large F-value indicates that most of the variation in the

Factor 1	Factor 2	Response		
		Specific heat (KJ/kg K)		Absolute average deviation
A: Temp K	B: Conc %	Actual	Predicted	
293.15	0.1	4	4.31745	14.1
333.15	0.1	6.3	4.48745	
293.15	0.6	4.5	4.31469	
333.15	0.6	8	4.48469	
284.87	0.35	4.1	4.27489	
341.43	0.35	7.5	4.51527	
313.15	0	3.9	4.409	
313.15	0.7	5.8	4.40513	
313.15	0.35	5.1	4.40707	
313.15	0.35	5.1	4.40707	
313.15	0.35	5.1	4.40707	
313.15	0.35	5.1	4.40707	
313.15	0.35	5.1	4.40707	
313.15	0.35	5.1	4.40707	

response can be explained by the regression model equation. Three-dimensional response surfaces generated from the predicted results Eq. 2 for specific heat of xanthan gum with temperature and concentration was shown in Fig. 4. The specific heat of xanthan gum solution was modelled through a linear multiple regression as a function of temperature (K) and concentration (X_x) as follows

$$Cp = 98.78575 - 0.6495T - 15.9955X_x + 0.06TX_x + 0.001109T^2 - 0.5X_x^2 \quad (3)$$

The predicted results of specific heat of xanthan gum solution using the quadratic equation generated by RSM were compared with the experimental values. The predicted value using Eq. 3 and the experimental results are shown graphically in Fig. 5.

It can be observed that the experimental and predicted results are in good agreement; the variation was confirmed using absolute average deviation (AAD). The absolute average deviation observed was 0.130 for the specific heat of xanthan gum.

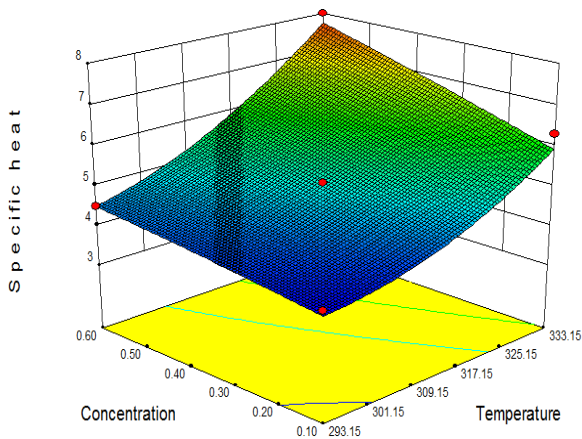


Fig -4: Three dimensional response of temperature and concentration influence on specific heat of xanthan gum solution

Goodness of fit by quadratic equation was checked using the regression coefficient R^2 . The specific heat of xanthan gum values of quadratic equation R^2 and adjusted R^2 value are found to be 0.9774, 0.9614 respectively.

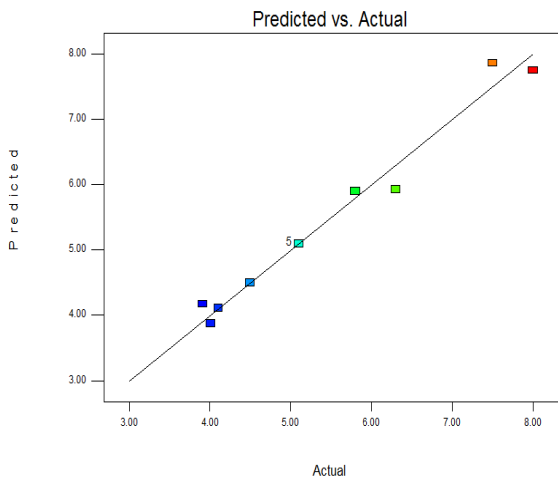


Fig -5: Specific heat of xanthan gum solution predicted results using equation 2 was compared with experimental results

Using ANOVA for the response surface, the significant effects of the operating variables were determined and the obtained results are shown in Table 4. The fisher F-values for specific heat of xanthan gum solution were higher representing that the model equation signifies the variation in the response.

Table -3: Experimental runs and comparison between the predicted result and the experimental result of specific heat of xanthan gum solution

Run	Factor 1	Factor 2	Response		
	A: Temp	B: Conc	Specific heat (KJ/kg K)		
	K	%	Actual	Predicted	Absolute average deviation
1	293.15	0.1	4	3.88	0.130
2	333.15	0.1	6.3	5.93	
3	293.15	0.6	4.5	4.50	
4	333.15	0.6	8	7.75	
5	284.87	0.35	4.1	4.11	
6	341.43	0.35	7.5	7.86	
7	313.15	0.00	3.9	4.17	
8	313.15	0.70	5.8	5.90	
9	313.15	0.35	5.1	5.1	
10	313.15	0.35	5.1	5.1	
11	313.15	0.35	5.1	5.1	
12	313.15	0.35	5.1	5.1	
13	313.15	0.35	5.1	5.1	

The model is statistically significant with larger F and p-values. The ANOVA of regression model observed for this quadratic equation being highly significant ($p < 0.0001$). The behavior of response surface in Fig. 4 also confirmed this implication.

CONCLUSIONS

The experimental results of the specific heat of xanthan gum obtained in this study have been analyzed for the influence of temperature and concentration of the specific heat of xanthan gum. The specific heat of xanthan gum solution measured by Joules calorimeter method revealed that it depends strongly on the concentration and temperature. An increase of specific heat of xanthan gum solution was observed, when both temperature and concentration increase. When compared to the specific heat of water, the specific heat of xanthan gum solution was higher.

The model equation proposed by semmar *et al.*, (2004) was tested using the experimental values of specific heat capacity of xanthan gum solution. Based on the equation proposed bysemmar *et al.*, (2004) using CMC and CPE solutions and the absolute average deviation found to be 14.1. A new empirical equation was developed to predict the specific heat of xanthan gum solution as a function of temperature and concentration using CCD along with RSM.

Table -4: ANOVA for response surface of the second-order polynomial equation for specific heat of xanthan gum solution

Source	The sum of Squares	df	Mean Square	F-Value	P-value Prob>F	Remarks
Model	18.84	5	3.77	60.79	< 0.0001	Significant
A-Temp	14.07	1	14.06	226.90	< 0.0001	
B-Conc	2.99	1	2.99	48.17	0.0002	
AB	0.36	1	0.36	5.81	0.0468	
A ²	1.37	1	1.37	22.10	0.0022	
B ²	0.01	1	0.01	0.11	0.7503	
Residual	0.43	7	0.06			
Lack of fit	0.43	3	0.14			
Pure error	0.00	4	0.00			

The new model equation obtained for estimating the specific heat of xanthan gum solution using response surface methodology was tested with the experimental data with a regression co-efficient of 0.9774. The proposed empirical equation could be used for the estimation of specific heat of xanthan gum solution for any combination of concentration and temperature.

NOMENCLATURE AND UNITS

C _p	Specific heat (KJ/kg K)
T	Temperature (K)
T ₀	Temperature of pure water at 273.15 (K)
X	Concentration (%)
M	Mass (g)
I	Current (amp)
E	Voltage (V)
D	Coefficients of concentration
A & B	Coefficients of temperature
t	Time (sec)
W	Weight (g)

Subscripts

x	Xanthan gum solution
i	Initial
f	Final
m	Copper material

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A. Arunagiri, is working as Assistant Professor in the Dept. of Chem. Engg., National Institute Of Technology, Trichy, Tamil Nadu, India, from 2006. He also worked as a Lecturer at A. C. College of Technology, Anna University, Chennai, Tamil Nadu, India. His research interests are Transfer Operations, Environmental Engineering and Multiphase flow. Dr. A. Arunagiri is a life member of Indian Institute of Chemical Engineers (LM-34030) and was awarded for Teaching Research Associate fellowship and Senior Research Fellowship by Anna University and CSIR respectively



Rudhra Anandandid his B. Tech in National Institute of Technology, Trichy, Tamil Nadu, India and belongs 2009-2013 batch. He coordinated Placement for 2010-2014 batch and worked as a manager for Delta-T (Heat Exchanger Design Event) of Pragyan (International Techno-Management festival of NIT, Trichy) in the year of 2012.

BIOGRAPHIES



K. Muthamizhi graduated as a Pharmaceutical engineer in 2007 from Bharathidasan Institute of Technology, Bharathidasan University, Tamil Nadu, Tiruchirappalli, India. She received the master degree in Chemical engineering from

Coimbatore Institute of Technology in 2010, Coimbatore and she is now pursuing her Ph.D. degree in National Institute of Technology, Tiruchirappalli and working as a Senior Research Fellow in CSIR project-NIT-Trichy under the supervision of Dr. P. Kalaichlevi.



Dr. P. Kalaichlevi, is working as Associate Professor in the Dept. of Chem. Engg., National Institute Of Technology, Trichy, Tamil Nadu, India. She received CSIR-SRF fellowship (1996-1998) for Ph.D Program and BOYSCAST fellowship 2004-2005,

from Department of Science and Technology, India She has undergone a research training program in National University of Singapore, Singapore under BOYSCAST