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THERMO-PHYSICAL PROPERTIES OF BEESWAX

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ABSTRACT
This study presents an investigation conducted on thermo-physical properties of beeswax. Bees wax has been identified with the potentials of being used in thermal storage application, its properties were said to differ according to nesting ecology. This research investigated the thermo-physical properties of three samples of beeswax from different nesting ecology using METTLER TELODO STAR^e SW13.00 version differential scanning calorimetry, thermo-gravimetric analysis and differential thermal analysis. The results showed that, the melting points of sample A, B and C are 60^{o}C, 60.01^{o}C and 64.94^{o}C respectively. Thermal conductivities of the samples are 3.45 W/m^{o}K, 6.69 W/m^{o}K and 1.8W/m^{o}K, latent heat of fusion of 133.32 J/g, 200.01 J/g and 66.93J/g for sample A, B and C. Hence, from the results obtained, beeswax can be used in low temperature thermal storage applications.

Keywords: Ecology, Thermogram, Endothermic, Exothermic, Nesting

INTRODUCTION

Beewax is the creamy coloured substance that bee uses in building comb of their nest. Very pure beeswax is white, but the presence of pollen and other substances cause it to become yellow (FAO, 2010). Beeswax is low temperature organic Phase Change Material (PCM). The empirical formula for beeswax is $C_{15}H_{31}COOC_{30}H_{61}$. It consists of palmitate, palmitoleate, hydroxypalmitate, and oleate esters of long chain aliphatic alcohols (Ramnanan-singh, 2012).

The newly made beeswax is white in colour which changes with time to yellow, dark yellow or brownish. The structure of beeswax is crystalline which is also dictated by the storage. Beeswax does not fully dissolve in any solvents at room temperature, but upon heating above the wax melting point it is soluble and also in ethanol (Bogdanov, 2009). Beeswax is a multi -component complex material with over 300 different components. It is made up of esters of higher fatty acids and alcohols. Beeswax also contains small quantities of hydrocarbons, acids and other substances. In addition, approximately 50 aroma components have been identified (Bogdanov, 2009). Breed *et al*.,(2016) in their study on three species of bees; Bombini, Meliponini and Apini investigated the relationship between nesting ecology and the thermo-physical properties of beeswax. They used Differential Scanning Calorimetry and concluded that, although bees in these groups share many physiological and ecological characteristics but they also exhibit important thermo-physical differences. Mohammad and Saeed (2016) used Differential Scanning Calorimeter (DSC) to characterize the PCMs and to determine the latent heat of fusion, phase change (melting) temperature, specific heat capacity, the enthalpy as a function of temperature, and the study experimentally investigated the uncertainty of thermal characterization of PCMs by DSC analysis. Ruguo *et al*,, (2011) undertook thermal analysis on waxes of four different insect species and compare it with paraffin and canauba wax using DSC. Their findings indicated that DSC was qualitatively and quantitatively available for thermal analysis of insect wax. Masae *et al.*, (2014)in an effort to integrate waxes in to cotton fabrics characterized Beeswax and Paraffin using DSC specifically a PerkinElmer DSC7. They concluded that DSC produce results of thermo-physical properties with better accuracy than the traditional mercury surface measurement. The aim of this research was to investigate the thermo-physical properties of three samples of beeswax from different nesting ecology.

MATERIALS AND METHODS

Materials/Equipment

Differential scanning calorimetry (DSC), thermo-gravimetric analysis (TGA) and differential thermal analysis (DTA). Beeswax samples were collected from three (3) different nesting ecologies within the savannah zone of Nigeria. The beeswax samples were leveled as follows: Sample A (beeswax collected from Kano), sample B (beeswax collected from Taraba) and sample C (beeswax collected from Sokoto).

METHODS

Determination of Thermo-physical properties

Differential Scanning Calorimeter (DSC) – METTLER TELODO STAR^e SW13.00 version is an equipment used in thermal analysis. It's capable of measuring the temperature dependent properties of materials i.e. melting temperature, heat capacity, crystallization, glass transition temperature. The equipment can be either heat flux or power compensated type (UserCom 2000). In this section an experiment using DSC was employed to determine the melting point and latent heat of the bee wax samples, density of the samples was found using the cylinder method of density determination, while the thermal conductivities of the samples were determined by calculation using the results obtained from the DSC experiment (i.e. values of melting point and latent obtained and the configurations of the crucible), so also the thermal diffusivities.

Determination of Melting point and latent heat of the beeswax samples

This experiment was carried out using the differential scanning calorimeter (DSC), which is the most preferred equipment used in determination of temperature dependent properties of materials because of its efficiency and precision. The experiment was conducted at the NLNG Laboratory of Ahmadu Bello University Zaria.

7.1mg of sample A was weighed in a hermetic aluminium pan (crucible) using a sensitive electronic weighing machine. The lid was pierced with a pin and placed on top of the pan. The covered pan and lid where sealed by a sample press. The above steps were repeated with an empty pan that was used as a reference pan. The sealed pans were then placed on the sample tray with the reference pan occupying the reference slot. The furnace chamber was opened by removing furnace lid with a tweezer. The reference pan was carefully put on the left hand side and the sealed sample pan on the right hand side of the furnace. The power to the DSC was then turned-on and the "STAR^{e}" software on the desktop of the computer was double clicked to establish communication with DSC module, on filling-in the user name and password, the 'method' was selected on the Routine Editor that appears on the window. The temperature of the sample A was then calibrated and the calibration was accomplished by pressing the reset button which resulted in resetting the temperature, cooling medium and the model of crucible used.

In the case of this particular test, temperature range of 60°C - 300°C was selected for the heating of the sample, air was chosen as the cooling medium and 40µl Aluminum pan was used as the crucible. On another column on the Routine Editor, the samples Mass in the range 4mg -8mg and sample level were selected. 7.1mg and Sample A were entered and the experiment was allowed to run. The thermal analysis measurement was started from the temperature of 60°C until the temperature reached 300°C at a heating rate of 10°C /min. A logger was used to record the results of the experiments and then saved on the computer attached to the system.

The same procedure was repeated for 4.8mg of sample B and 4.7mg of sample C. The results obtained for the 3 beeswax samples are shown in figures 1a, 1b and 1c respectively.

Determination of Density

The density of the samples was determined using the mass density equation. The data was obtained using the cylinder method of density determination. A measuring cylinder was used to measure 352mL of water and 158.3g of beeswax was poured into the cylinder. A total volume of 520mL was obtained. The difference between the initial volume and the final volume gave the value 167.7 x 10^{-6}m^{3}. The density of the samples was determined using the following equation

$$\rho = \frac{M}{V} \qquad (1)$$

The same procedure was used to determine the density of the remaining samples.

Determination of Thermal Conductivity

The thermal Conductivities of the sample were determined using the following Fourier Equation (Zhao *et a*l., 2016).

$$\frac{Q}{t} = \frac{K \times A \times \Delta t}{X} \qquad (2)$$

$$\frac{Q}{t} = C_P \times heating\ rate \qquad (3)$$

Where,

$\frac{Q}{t}$ is heat per unit time (W), $\frac{\Delta t}{X}$ is temperature gradient (°K/m), K is thermal conductivity (W/m°K), A is Area of cross-section in (m^{2}) of the sample A

$$A = 2\pi R^2 + 2\pi RH \qquad (4)$$

Since the crucible is cylindrical in shape, the following formula was used to determine the value of R from the relation, $V = \pi h R^2$.

$$\text{Radius} = R = \sqrt{\frac{V}{\pi H}} \qquad (5)$$

where,

V is the volume occupied by the sample; H is the height occupied by the sample;

H_C = is the Height of the crucible$= 1.6mm$; the mass of the crucible = 8mg; Volume of Aluminium crucible = $V = 40\mu l = 4e^{-8}\, m^3$; thickness of aluminum crucible is $x = 0.47mm$

The change in temperature from the result of the analysis was obtained as; onset = 60°C, endset = 100.87°C; $\frac{Q}{t} = 22.22\ W$,

Therefore, the height occupied by 7.1mg in the crucible = $H = \frac{1.6 \times 7.1}{8} = 1.42mm$

The volume occupied by 7.1mg of the sample = $V = \frac{4 \times 10^{-8} \times 7.1}{8} = 3.55 \times 10^{-3} m^3$

Substituting the values of H and V in equation 4 and 5. The values of R and A are 2.801mm and A = $7.43 \times 10^{-5}\, m^2$ respectively.

The thermal conductivity of sample A is $3.45 W/m°K$ which was obtained using equation 1. The same procedure was used to determine the thermal conductivities of samples B and C.

Determination of thermal diffusivity

The thermal Diffusivity was obtained using the following equation given by (Yang 2002) $a = \frac{k}{\rho \times Cp}$ (6)

Where

K is the thermal conductivities of the samples; ρ is the density of a sample; C_p is the specific capacity of the samples

RESULTS

Melting point and latent heat of fusion of beeswax samples

Figures 1(a - c) are the results of differential scanning calorimetric used in the determination of some of the thermo-physical properties of the beeswax, namely, the melting point and latent heat of fusion. The thermogram is a plot of heat gained or lost by the beeswax against temperature, the area under the curve represents the amount of heat involved in the melting of the material. It can be seen from the graph that it is an endothermic process, where the material absorbs heat from the surrounding and store it as latent heat during the melting process. Due to sensitivity of the DSC some other peaks were present in the thermogram which also proves the multicomponent nature of beeswax as postulated in literatures. It could be observed that the peak was highest in the temperature range of 90°C -100°C in all the three samples which was an indication of the importance of that range in handling the material. The melting points of the three samples A, B and C are 60°C, 64.94°C and 60.01°C respectively, similarly the latent heat of the samples are 133.32J/g, 200.01J/g and 66.93J/g.

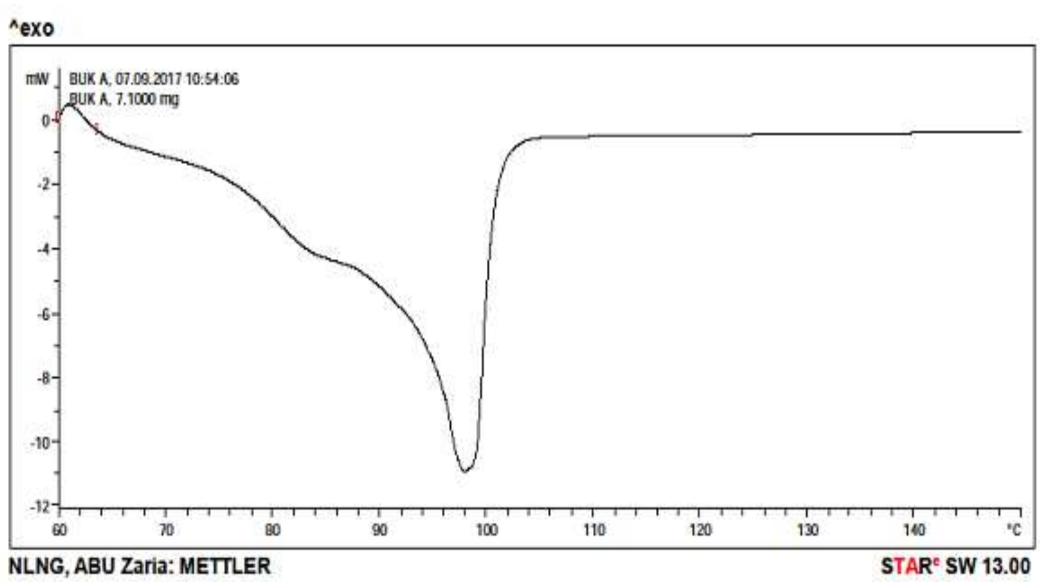

Fig. 1 (a): Thermogram of sample A from DSC experiment

In figure 1 (a) on heating the 7.1mg of the sample at 10°C/min it continues to absorb heat in the form of sensible heat without change in phase, it could be seen from the thermogram that at the temperature of around 60°C to 62°C there is a little exothermic region up to 0.5mW, thereafter there is gradual slope in the endothermic region to an energy value of -6mW up to around 90°C, from there to 98°C there is steep slope that represent highest point of energy absorption to -11mW. At around 99°C to 100°C there is sharp rise in the thermogram with energy value -11mW to -1.5mW and from 102°C up to around 150°C the value of energy absorbed was kept almost constant i.e. -1.5mW to -0.4mW. The region under the graph is the amount of sensible heat absorbed by the beeswax.

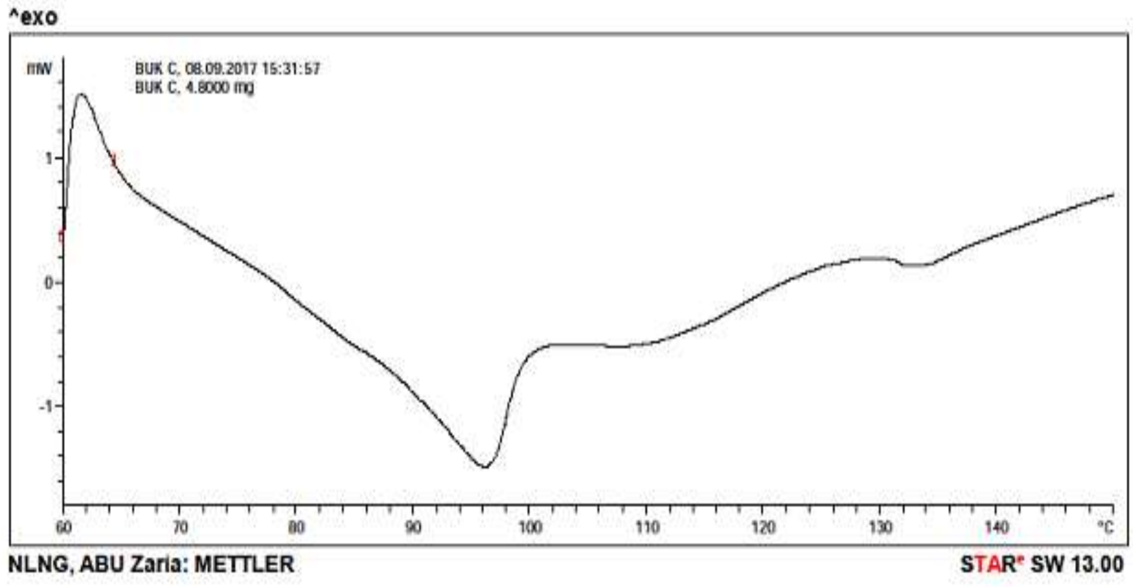

Fig. 1 (b): Thermogram of sample B from DSC experiment

In figure 1 (b) on heating the 4.7gm of the sample at 10°C/min it continues to absorb heat in the form of sensible heat without change in phase, from 60°C at 0 mW there was gradual slope in the endothermic region up to around 94°C with energy value -6mW, from there to 97°C there is steep slope that represent highest point of energy absorption to a value of -12mW. At around 99°C to 100°C there is sharp rise in the thermogram with energy values of -12mW to -1mW and from 100°C up to around 150°C the value of energy absorbed was kept almost constant as in the previous sample with energy value of -2mW to -1.5mW. The region under the graph is the amount of sensible heat absorbed by the beeswax.

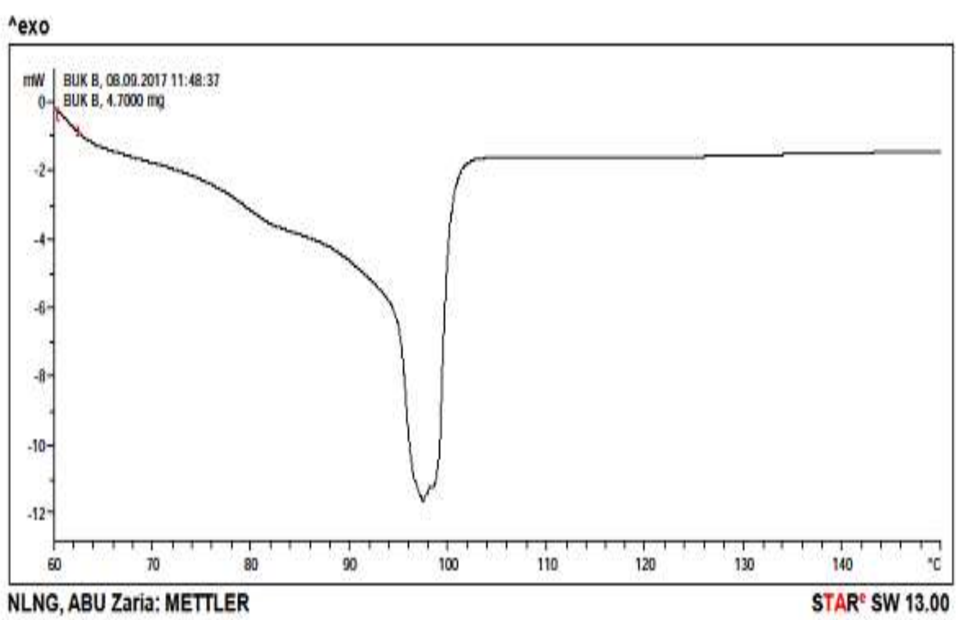

Fig. 1 (c): Thermogram of sample C from DSC experiment

In figure 1 (c) on heating the 4.8gm of the sample at 10°C/min it continues to absorb heat in the form of sensible heat without change in phase, there is a peak in the exothermic region up to around 62°C, from 62°C there is gradual slope in the endothermic region up to around 96°C from there to 100°C there is steep slope up to – 0.5mW and from 100°C up to around 150°C the value of energy absorbed rises gradually up to 150°C the energy value changes from -0.5mW to 0.8mW. The region under the graph is the amount of sensible heat absorbed by the beeswax.

Table 1: Thermo-physical properties of Beewax

Physical properties	Sample A	Sample B	Sample C
Transition Temperature(^oC)	60	64.94	60.01
Latent Heat (kJ/kg)	133.2	200.01	66.93
Thermal Conductivity(W/m^{o}K)	3.45	6.69	1.8
Thermal Diffusivity(m^2/s)	$1.65x10^{-4}$	$2.08x10^{-4}$	$1.72\ x10^{-4}$
Density (kg/m^3)	943.9	962.76	971.74

DISCUSSION OF RESULTS

Thermo-Physical Properties of Beeswax

Table 1 shows the experimental and calculated results of thermo-physical properties of beeswax. The melting temperatures obtained from the DSC are 60.00°C, 64.94°C and 60.01°C for samples A, B and C respectively, similarly in the same order the latent heat were 133.32 J/g, 200.01 J/g and 66.93J/g. From these results and thermograms it could be observed that the melting temperature of all three samples falls within the range of theoretical value of the melting temperature of beeswax as obtained by other researchers; Dinker *et al*., (2017b) obtained 62°C as melting point and 186kJ/kg as the latent heat, Attama *et al*., (2006) obtained 63.6°C as melting point, Wi et al., (2017) obtained 61.05°C as melting point and 173.6J/g as latent heat, similar results for melting point in the range of 59°C - 64°C and latent heat in the range of 140J/g – 200J/g were obtained by (Bal et. al, 2010; Sharma et al., 2009; Cheng et al., 2018; Amin et al., 2017; Sinaringati et. al., 2016). Sample B shows high latent heat which is also an indication of its high thermal mass and superiority in terms of thermal energy storage. The latent heat of sample C was found to be the lowest with 66.93J/g and out of the theoretical values cited, this may be due to impurities in the sample, the specie of the bee or its nesting ecology. Due to sensitivity of the DSC some other peaks were present in the thermogram which also proves the multicomponent nature of beeswax as postulated in literatures. The highest amount of heat flow was observed within the range of 90°C -100°C in all the three samples which is an indication of the importance of heating the sample up to that temperature level to store more energy. The variation in latent heat may be due to the postulation that nesting ecology or the purity of the samples has direct effect on thermal properties of beeswax (Breed, 2016). DSC studies indicate that beeswax melts over a relatively wide temperature range, with the onset of melting occurring at a temperature below the temperature at which melting is first observed (Breed, 2016). Thermal conductivities of sample A, B and C respectively are 3.45 W/m^{o}K, 6.69 W/m^{o}K and 1.87W/m^{o}K as obtained through data analysis of the DSC results of melting point and latent heat. These findings implied that there may be intra or inter variation of thermo-physical properties among the species of beeswax based on their nesting ecology, and that beeswax can be used in low temperature heat storage application especially those with high latent heat which is the key determinant of heat storage capacity of the materials. These results might be challenged by the inability of the researcher to collect the beeswax from the same species of bee wax but rather resorted to restricting the collection on nesting ecology. Other limitations that might affect the comparison of results is the masses of the sample used which apparently did not follow any order but rather the values were selected within a range given by the equipment producers (i.e. 4mg – 8mg), and, yet the results obtained have provided a reasonable evidence to show the effect of this variation. Sample A with the highest sample mass has lower value of latent heat and melting temperature than sample B whose sample mass was the least and sample C whose sample mass was higher than Sample B but lower than sample A has the least value of latent heat and higher value of melting point than sample A.

CONCLUSION

The thermo-physical properties are the temperature dependent properties of a material. Thermo-physical properties of three un-identical samples of beeswax were investigated using DSC experiment and data analysis. The results of the investigation showed a melting point of 60°C, 64.94°C and 60.01°C; latent heat of 133.32J/g, 200.01J/g and 66.93J/g; thermal conductivity of 3.45 W/m^{o}K, 6.69W/m^{o}K and 1.8W/m^{o}K; thermal diffusivity of 1.6 $x10^{-4}$ m^2/s, 2.08 m^2/s and 1.72 $x10^{-4}$ m^2/s for sample A, B and C respectively. From the results it can be concluded that all the three samples have shown the capability of being used in low temperature applications but sample B has shown superior properties for its higher latent heat of 200.01J/g and higher melting point 64.94°C.

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