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Proceedings Study and characterization of phase change material-recycled paperboard composite for thermoregulated packaging applications ⁺

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Abstract: Beeswax is a bioderived phase change material (PCM) with phase change temperature 12 around 60°C which is suitable for thermoregulating hot served food packages. Beeswax should be 13 shape stabilized for thermoregulation purpose. This report has used recycled paperboard as matrix 14 for shape stabilizing beeswax. Beeswax showed melting enthalpy of 216.09 J/g and melting enthalpy 15 of composite with beeswax content 45% was 102.51 J/g. Thermal conductivity of beeswax and com-16 posite with 45% beeswax calculated with T-history method as 0.285 and 0.157 respectively. To ad-17 dress concerns such as toxicity, environment friendly nature and recycling, beeswax-recycled pa-18 perboard composite should be considered as promising candidate. 19

Keywords: Phase change material; thermal energy storage; beeswax; latent heat; packaging

1. Introduction

Hot served food items are maintained around 60°C temperature. Delay caused due 23 to interventions in supply chain activities reduce temperature of food products. Such 24 products can be complemented with phase change material (PCM) pouches which assure 25 temperature maintenance. PCMs absorb large amount of heat at its phase transition tem-26 perature and release heat to cooler environment to establish hot food temperature. Many 27 research groups have used PCM in delivering temperature sensitive food products. Wang 28 et al. used [1] microencapsulated PCM stored in zip lock bag to transport meat package 29 from plastic crisper while maintaining temperature around 4 °C. Energy consumption of 30 reefer transported food items reduced by combining PCM into walls of vehicle [2-4]. Such 31 composite structures are suitable for transporting food items without refrigeration in 6-9 32 hours' time. Combination of vacuum insulation panel and PCM panel in small container 33 package can maintain temperature of 2-8 °C for 72 hours without refrigeration system [5]. 34 Even hot beverage cups maintain sipping temperature for longer duration with PCM [6]. 35

In this chapter, a PCM composite was prepared with reusable, biodegradable materials. The simple method of preparation makes it suitable for low cost commercial applications. The prepared PCM composite assure temperature maintenance of food item for longer time in food delivery process while utilizing lesser amount of energy. 39

2. Materials and Methods

2.1. Materials

Beeswax procured from anahaTM. Commercially available paper board obtained 42 from local market. Tween 80 was obtained from SD fine chemicals private limited. Sodium 43

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Copyright: © 2021 by the authors. Submitted for possible open access publication under the terms and conditions of the Creative Commons Attribution (CC BY) license (https://creativecommons.org/license s/by/4.0/). benzoate was purchased from shree lakshmi chemicals. DI water was used for 1 experimental work. Four channel temperature data logger was assembled and 2 programmed with Arduino UNO. Two cartons of dimensions 19x13x25 cms were used. 3

2.2. Method of preparation

2.2.1. Paper pulp preparation

Paperboards were cut into pieces and boiled water added to these pieces to prepare 5% solution. Solution was kept overnight to obtain slurry. The slurry was mechanically grinded.

2.2.2. Beeswax dispersion preparation

Beeswax in the quantity of 5 gm was melted at 70 °C. 95 ml of boiled water added to molten wax. Two drops of tween 80 emulsifier and 2% antimicrobial agent sodium benzoate added to the solution. The mixture was stirred for 30 minutes at 70 °C on magnetic stirrer.

2.2.3. Beeswax dispersion preparation

Prepared paper pulp added to beeswax dispersion and stirred for 30 minutes at 70 15 °C. Quantity of pulp varied to prepare dispersion with 0% 20% 40% 60% and 80% beeswax 16 concentration. Prepared dispersion filtered with filter paper of pore size 8 to 10 micron. 17 The suspension was filtered by vacuum filtration with Buchner funnel. The material re-18 mained above filter paper was peeled off and kept in silicone mould of 9x9x0.2 cm size. It 19 was then dried for 7 days and hot pressed with fabric covering. The highest concentration 20 of beeswax showing no leakage was determined. Three composites were prepared in-21 creasing beeswax concentration in steps of 5%. These samples will help to determine high-22 est quantity of beeswax that can be incorporated in paper. 23

2.3. Characterization techniques

2.3.1. Leakage test

One-two grams of composite material and small amount of tissue paper filled in test tube. Test tube was heated to 70 °C. Weight of paper before and after heating was tested. If weight increases, it indicates leakage from composite.

2.3.2. T-History method

The experimental setup consisted of two identical test tubes consisting equal amount 30 of glycerine (reference) and sample PCM, heating chamber and temperature sensors. The 31 sample and reference were heated to 70°C and allowed to cool down to room temperature. 32 There temperature drop in reference and sample in the temperature range 70°C to 27°C 33 was recorded. These values were used to plot temperature-time curves of the PCM sample 34 and reference. 35

The specific heat and thermal conductivity of the solid PCM are determined using the following formula

$$C_{p} = \frac{m_{R} * c_{p R} + m_{t} * c_{p t}}{m_{p}} \times \frac{A_{p}}{A_{R}} - \frac{m_{t} * c_{p t}}{m_{p}}$$
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where m_R is mass of reference material, m_t is mass of tube material, m_p is mass of 39 PCM, c_p is specific heat capacity of PCM sample, $c_p R$ is specific heat capacity of reference material, $c_p t$ is specific heat capacity of tube material, A_p is area under the cooling 41 curve of PCM and A_R is area under the cooling curve reference material. 42

Thermal conductivity of the solid PCM was calculated using the formula

$$k_{p} = \left[1 + \frac{c_{p}(T_{m} - T_{\infty})}{H_{m}}\right] / 4 \left[\frac{t_{f}(T_{m} - T_{\infty})}{R^{2}\rho_{p}H_{m}}\right]$$

$$44$$

where, k_p is the effective thermal conductivity of the PCM, c_p is the specific heat of 45 the PCM, ρ_p is the density of the PCM, R is radius of test tube, H_m is the heat of fusion 46 of PCM as obtained from the DSC results and $T_m \& T_\infty$ is the temperature of melting and 47 atmosphere respectively. Time of solidification of the molten PCM is denoted by t_{f_1} . 48

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2.3.3. Heat release performance in carton

PCM composite sheets were placed inside carton of larger size with insulation foam. 2 Smaller size carton was placed into it. 50ml of boiled water was placed inside smaller 3 carton. A control sample prepared with same procedure without PCM was also tested the 4 same way. Temperature in center of the cartons and outside temperature was measured 5 with temperature data logger. The arrangement can be better understood with figure 1. 6



PCM-Recycled paper sample

Control sample

Figure 1. Assembly of sample for heat release performance testing

2.3.4. Differential scanning calorimetry

About 5 mg of sample was used for the analysis. To remove the thermal history of 10 sample, sample was heated from room temperature to 200 °C at a heating rate of 10 °C/min 11 under a nitrogen atmosphere. The sample was held at 200 °C for 2 minutes. The crystallizing and melting characteristics of sample were analyzed in the next scan. Differential 13 scanning calorimetry (DSC) scanning was performed from 0 °C to 200 °C at a heating rate of 10 °C/min under a nitrogen atmosphere. 15

3. Results

Samples with 20% and 40% beeswax concentration showed negligible leakage. Samples with 60% and 80% beeswax concentration showed 12.66% and 20.38% increase in 18 weight of paper respectively. Samples of composite with 45%, 50% and 55% beeswax content were made following the previously mentioned method. Leakage tests showed 3.83% 20 and 9.25% weight increase for 50% and 55% beeswax containing composites. Negligible 21 leakage was obtained with 45% beeswax-recycled paper composite. 22

As shown in temperature-time curve in figure 2, beeswax content has larger area under the curve depicting highest heat storage capacity. Thermal conductivity of beeswax and composite samples with 45%, 40% and 20% beeswax calculated as 0.285, 0.157, 0.141 25 and 0.119 respectively. These values are comparable to observed values in literature [7]. 26



Figure 2. Temperature-time curve of beeswax and beeswax composite

Figure 3 gives temperature-time profile of carton containing beeswax composite and control sample of carton. Composite assembly took 6823 seconds for reaching ambient temperature. Control assembly took 6259 seconds to reach ambient temperature. Initially 5 PCM temperature drops around 38ºC due to absorption of heat by PCM. This temperature 6 7 was lesser than control assembly. But at 2362 second, temperature started to increase with heat release from PCM. After that temperature slowly reached ambient temperature. 8



Figure 3. Temperature-time profile of control sample, composite sample and ambience

Beeswax showed melting enthalpy of 216.09 J/g and melting enthalpy of composite 11 with beeswax content 45% was 102.51 J/g. The DSC thermograms are given in figure 4. 12

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Figure 4. DSC thermogram for melting process

4. Discussion

With increasing content of beeswax, enthalpy of phase transition increases. This implies increase in thermoregulation capacity is possible with high beeswax loading in recycled paper composite. But, the limit of maximum beeswax incorporation in recycled paper composite is constrained by leakage. Leakage test indicated that samples with 60 and 80% beeswax content showed severe leakage which is not suitable in practical applications. Pores of paper cannot prevent leakage of PCM above its melting point. Thus, 45% beeswax is the highest concentration that can be incorporated in paperboard with mentioned technique.

As the beeswax concentration decreases, the conductivity decreases. The beeswax 12 filled inside the pores of paper which otherwise filled with air. That is why increase in 13 beeswax concentration increases thermal conductivity of composite structures. With de-14creasing beeswax content, area under the temperature-time curve reduces. This means 15 that the heat is dissipated at faster rate when paper content in composite is higher. But, 16 composite assembly kept the inside temperature at higher value for more time than con-17 trol assembly. This higher temperature inside the carton is from the heat release from PCM 18 to inside chamber. This phenomenon can be used to maintain higher temperature in case 19 of delayed delivery. 20

5. Conclusions

Optimization study revealed that 45% is the maximum amount of beeswax that can 22 be incorporated in paper composite. Thermal conductivity of composite increases with 23 increase in beeswax concentration. The carton containing PCM sheets maintain tempera-24 ture at higher level for longer time than for carton without PCM. Phase transition temper-25 ature of prepared composite is around 60°C. If food items once prepared and stored in 26 container equipped with beeswax-recycled paper composite, it can maintain temperature 27 of food item for longer time in food delivery process while utilizing lesser amount of en-28 ergy.

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I	Data Availability Statement: The data presented in this study are available on request from the	1
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