Original Article

Composite Phase Change Material for Improving Thermal Protection Performance of Insulated Packaging Container

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Abstract — Rampant growth in fast food consumption translated into the emergence of quick home delivery services. These services use insulated containers for delivering hot foods. However, such containers are not helpful for longer journey hours. Inclusion of latent heat storing and form-stabilized phase change materials (PCMs) in such containers is an effective way of maintaining the required temperature for longer hours. This study used the recycled paper board as the matrix to form-stabilize biobased PCM beeswax. SEM analysis confirmed infusion of beeswax into the fibrous mesh of recycled paper. DSC analysis of composite showed good heat storing capability with melting enthalpy of 102.51 J/g at peak phase transition temperature of 59.92 °C. A model of insulated packaging container with composite PCM was constructed. This model maintained the temperature of the packaged food in the designed period of two hours. The fabricated composite can fulfil the need to use environment-friendly packaging material.

Keywords — *Phase change material, Thermal energy storage, Beeswax, Microencapsulation.*

I. INTRODUCTION

Heat regulation performance of Phase Change Materials (PCMs) has been found useful in various devices such as solar water heaters [1], packed bed Thermal Energy Storage (TES) devices [2], household refrigerators [3], solar cookers [4], and solar power plants [5]. PCM absorbs a substantial quantity of heat at their phase transition temperature to maintain the product's temperature at a constant level. When the product's temperature drops below the phase transition temperature of PCM, PCM releases back stored latent heat to increase the product's temperature. The addition of latent heatstoring PCM in cold-stored food and pharmaceutical products packaging has increased temperature retention time [6], [7]. The commonly used arrangement for PCM incorporated packaging containers can be studied in figure 1. The inner carton contains the thermally sensitive product. Six PCM plates surrounded the six faces of the inner carton. These plates were further combined with insulated material from six sides. This assembly was then

placed in the outer carton for ease of transportation. Insulation avoids heat release to the outer environment. The combined effect of insulation and PCM ensures product temperature maintenance at the desired level for longer hours. The time of temperature regulation depends on the weight of PCM. The prefabricated PCM plates can maintain temperature for a longer time than required with the expense of high packaging weight.



Fig. 1 Arrangement for PCM incorporated packaging container

In the era of fast food consumption, the home delivery services transporting hot food to the consumer's doorstep are increasing. Traditional supply chain systems suffer from delays in the delivery process due to many unavoidable circumstances. Delay in the delivery results in cooled food, which disappoints consumers. The majorly used present systems deploy insulated containers for delivery which are inadequate to maintain temperature for longer journeys. PCMs with phase transition temperature above ambient temperature has been used in pizza containers [8], cups [9], dishes [10], and thermos [11]. Special containers were fabricated to engulf PCM inside containers, as shown in figure 2. The space between the inner and outer bodies was filled with PCM. The PCM layer acts as a thermal buffer and maintains the food temperature near the phase transition temperature of PCM. But, process modification for preparing such containers increases the cost of production. Thus, there is a need for simpler packaging designs for PCM incorporated containers.



Fig. 2 Arrangement of PCM incorporated containers serving hot food and beverage

The PCMs used in various applications were generally petroleum products. Increasing efforts to minimize carbon footprint demands for biobased substitutes. Beeswax is a biobased PCM with phase change temperature roughly in the range 57-63 °C [12], making it ideal for thermoregulating hot food packaging. This study used a simple PCM form-stabilization process to maintain food at a higher temperature. For form-stabilization, the beeswaxrecycled paperboard composite was fabricated. The fibrous mesh structure of recycled paperboard hinders leakage of latent heat-storing molten beeswax. The composite PCM sheet weight can be adjusted as per the time required for thermoregulation. The custom choice of PCM weight reduces packaging weight and the cost of transport. The temperature maintenance time of the form-stabilized composite was measured with a prototype model.

II. EXPERIMENTAL DESIGN

A. Materials

AnahaTM beeswax was used. Unbleached paperboard available in the local market was utilized. SD fine chemicals private limited provided Tween 80. Shree Lakshmi chemicals supplied the antimicrobial agent sodium benzoate. For the experiments, deionized (DI) water was utilized. Arduino UNO-based temperature data logger assembled with LM 35 temperature sensors. The temperature measurement accuracy of the data logger was 0.1 °C. Temperature measurements were performed with the data logger. Expanded polystyrene (EPS) insulation foam sheet of 13 mm thickness was purchased. Two larger paperboard cartons with 250x190x130 mm and two smaller paperboard cartons with 220x160x100 mm dimensions were purchased from the Rohanpack private limited.

B. Preparation method

The composite fabrication process consisted of six steps, as depicted in figure 3. Firstly, a paper pulp solution

with five weight percent concentration was made by soaking chopped paperboard pieces overnight in hot water. The prepared slurry was ground to defragment paperboard to individual fibre levels. The second step was the preparation of five weight percent beeswax dispersion. Beeswax was melted at 70 °C. DI water, one weight percent emulsifier, and two weight percent antimicrobial agent were added to the molten wax. On a magnetic stirrer, the mixture was stirred for 30 minutes at 70 °C. The emulsifier helps to stabilize molten beeswax particles in the aqueous medium. The paper pulp solution was added to beeswax dispersion in the third step. Different mixtures were made with the final concentration of beeswax as 20%, 40%, 60%, and 80%, respectively. It helped to determine the range of beeswax concentration that can be incorporated in composite with minimum leakage. The fourth step involved homogenizing prepared mixtures on a magnetic stirrer for 30 minutes at 70 °C. The fifth step involved vacuum filtration with a Buchner funnel. Filter paper with pore sizes of 8 to 10 microns was used to filter the prepared dispersion. The material above the filter paper was peeled off and converted to sheets. It was then airdried for 48 hours.

C. Leakage test

In a test tube, two grams of composite material and a little piece of tissue paper were inserted. The temperature of the test tube was increased to 70 °C and maintained for 10 minutes. The weight of the paper was measured before and after heating. Percentage weight difference after heat treatment was considered as leakage percentage. The concentration of beeswax with the least percentage of leakage was found. Three composites were made with beeswax concentrations increasing by five weight percent each time. These samples will aid in determining the maximum amount of beeswax that may be used in the paper composite. The leakage percentages of newly prepared samples were calculated. The beeswax concentration with the lowest leakage percentage was considered as optimized concentration.

D. Morphological analysis

Morphological interaction between beeswax and fibres was tested with FEI Quanta 200 Scanning Electron Microscope (SEM).

E. Differential scanning calorimetry

Latent heat storage enthalpy and phase transition temperature of beeswax and beeswax composite can be found with differential scanning calorimetry (DSC) analysis. The sample utilized for the analysis was about 5 mg. The sample's thermal history was eliminated by heating in a nitrogen environment from room temperature to 200 °C at a rate of 10 °C/min. The sample was kept at 200 °C for 2 minutes. The following scans were performed to understand the sample's crystallizing and melting properties.



Fig. 3 Composite preparation process

F. R-value

The ice-melt test was used to compute the R-value of the insulated container [13]. R-value provides information about the resistance to heat transfer. The R-value determination process involved placing a known quantity of ice inside the insulated container and measuring the amount of ice melted. The melt rate was calculated by the following formula:

$$R = \frac{A \times \Delta T}{M \times \Delta H}$$

Where,

R - Heat resistance value of the insulated box

A - Surface area inside the box

 ΔT - Temperature difference between the ambient air and phase transition temperature of ice

M - Weight of water collected divided by time of testing ΔH - Latent heat enthalpy of ice

G. Estimation of the amount of composite

The heat storage density of PCM depends on the amount of PCM. The amount of PCM composite required to maintain temperature for the desired period can be calculated from the following equation:

$$M = \frac{A \, x \, \Delta T}{R \, x \, \Delta H}$$

Where,

M - Melt rate of PCM composite

A - Surface area inside the box

 ΔT - Temperature difference between the ambient air and phase transition temperature of PCM composite *R* - Heat resistance value of the insulated box ΔH - Latent heat enthalpy of PCM composite

H. Heat release performance in cartons

The EPS sheets were placed near the inner wall sides of the larger carton. PCM composite sheets were placed adjacent to EPS sheets on six sides. A small carton was inserted into this assembly. Figure 4 illustrates the configuration better. The smaller carton was filled with 50 ml of boiling water container. The same process was used to create the control sample without PCM composite sheets. The arrangement was as shown in figure 4. The temperature data recorder monitored the temperatures in the middle of the smaller cartons and the outside ambient temperature.



PCM Composite Sample

Control Sample

Fig. 4 Heat release testing in cartons

III. RESULTS AND DISCUSSION

A. Leakage test

The results of leakage tests are presented in table 1. There was no leakage in samples containing 20% and 40% beeswax, respectively. Paperweight increased by 12.66% and 20.38% in samples with 60% and 80% beeswax concentrations, respectively. So, composite with 40% beeswax concentration showed negligible leakage. To find beeswax composite with the least leakage level, samples of composite with 45%, 50%, and 55% beeswax content were created. Leakage tests revealed a 3.83% and 9.25% weight increase for composites having 50% and 55% beeswax, respectively. With a 45% beeswax-recycled paper combination, there was no leakage. The phase transition enthalpy increases as the amount of beeswax in the mixture increase. This means that a high beeswax loading recycled paper composite in the can boost thermoregulation ability. However, leakage limits the amount of beeswax incorporated in a recycled paper composite. Leakage tests revealed that the sample containing 45% beeswax concentration is fit for practical use.

Sample	Leakage (%)
20% beeswax	0.34
40% beeswax	0.12
45% beeswax	0.85
50% beeswax	3.83
55% beeswax	9.25
60% beeswax	12.66
80% beeswax	20.38

 Table 1. Leakage characteristics of composites

B. Morphological analysis

Paper is made of cellulose fibres. The mesh structure created by fibres makes paper porous. These micrometre size pores can be observed in figure 5(a). In the SEM figure 5(b) of recycled paperboard composite with 45% beeswax concentration, pores of paper were seen filled. This feeling material is beeswax. When the prepared composite was heated well above the phase transition temperature of beeswax at 70 °C, beeswax did not leak away. The micrometric pores hinder leakage by engulfing small domains of beeswax. Paper pores prevent PCM from leaking over its melting point. SEM images helped to understand this morphology of composite.



Fig. 5 SEM image of (a) paper (b) recycled paperboard-beeswax composite

C. Differential scanning calorimetry

Beeswax is a mixture of long-chain fatty acids and esters [14]. These long-chain compounds absorb a large amount of latent heat and change from solid to liquid state. When beeswax is trapped in microporous paper, the crystalline structure of beeswax undergoes the phase change process in confined space. The microdomains of crystal change structure to the amorphous phase in response to heat absorption [15]. Structural differences associated with liquid beeswax and amorphous beeswax state caused changes in thermal properties. Beeswax has a melting enthalpy of 216.09 J/g, whereas a composite containing 45% beeswax has a melting enthalpy of 102.51 J/g. Figure 6 depicts the DSC thermograms. A lesser amount of latent heat was required to achieve the amorphous phase, and a larger amount of latent heat was required to attend the free-flowing liquid phase. The structure of the material depends on temperature. Reduction of phase change temperature of composite to 59.92 °C from 61.11 °C phase change temperature of beeswax confirms this phenomenon. Cellulosic fibres of paper do not possess any structural change in the phase transition of beeswax. The blending of beeswax with the matrix of recycled paperboard gives the dual advantage of TES and leakage prevention.



Fig. 6 DSC thermograms of paper, PCM composite, and beeswax

D. R-value

The fluted cardboard sheets and EPS sheets insulate packaged products from the outer environment. The extent of insulation is given by R-value. The R-value of the present system is calculated as follows

$$R = \frac{A \times \Delta T}{M \times \Delta H}$$
$$= \frac{[2(21.6 \times 15.6) + 2(21.6 \times 15.6) + 2(21.6 \times 15.6)] \times 24 \times 60 \times 60}{0.00513 \times 335}$$

$$= 1.72 \text{ m}^2 \text{ °C} / \text{W}$$

The R-value is directly proportional to the inner surface area of the package and the temperature difference between ice and ambience. The latent heat absorbed by ice in test time for converting to liquid water was represented in the denominator. The R-value of $1.72 \text{ m}^2 \text{ °C/W}$ indicates good insulation. The R-value helps to calculate the amount of PCM composite required in packaging assembly.

E. Estimation of the amount of composite

For a long time of temperature maintenance, a large amount of PCM composite is required. The amount of composite PCM depends on the inner area of the package, the temperature difference between ambient temperature and phase transition temperature of composite PCM, Rvalue, and latent heat of phase transition. The following formula can calculate the amount of composite PCM required:

$$M = \frac{A \times \Delta T}{R \times \Delta H}$$
$$= \frac{[2(21.6 \times 15.6) + 2(21.6 \times 15.6) + 2(21.6 \times 15.6)] \times 36 \times 60 \times 60}{1.72 \times 102.51}$$

= 176.17 g/h

$$= 352.34 \text{ g/2h}$$

For a journey of 2 hours, 352.34 g of PCM composite can maintain temperature near phase change temperature of beeswax composite, i.e., 59.92 °C. The paper stack structure of prepared composite PCM sheets helps in using the exact amount of PCM in package design. The commercially used PCMs come in plate structures in predesigned weights. The resultant weight of plates is mostly higher than required. The paper sheets can be easily distributed on all sides of the package in precise quantity as required in the package.

F. Heat release performance in cartons

The theoretically calculated temperature maintenance time is 2 hours. The assumption made during calculation is that the PCM composite is fully charged with heat at 65 °C. The real-time performance of composite PCM can be calculated in the carton test. In the carton test, the PCM composite sheets were charged at 65 °C in the oven for 30 minutes. The temperature profile inside the small carton in the PCM composite sample and control sample can be observed in figure 7. For the first nine minutes, the temperature of both samples showed a decrease in temperature in a similar way. Slowing down temperature becomes a slow process in composite PCM samples. At 87th minute, the temperature reached 60 °C. The temperature change from seventh to 87th minutes was reduced with latent heat absorption from PCM composite. Temperature lag in reaching phase change temperature can be associated with the air-filled fluted cardboard wall. It reduced the heat transfer rate.

On the other hand, the control sample observed a constant decrease in temperature to achieve ambient temperature at 66th minute. The objective of our study is to maintain food at higher temperatures in transportation time. The insulation alone cannot sustain food temperature to a higher level for a longer duration. The EPS insulation cannot resist heat from escaping to the atmosphere. The large phase change enthalpy at 60 °C maintains the temperature inside the carton for two hours. The

experimental values of temperature match theoretically calculated temperature values.



Figure 7. Temperature-time profile of control sample, composite sample, and ambience

IV. CONCLUSIONS

A PCM composite was made with reusable, biodegradable materials. It is suited for low-cost commercial applications due to its easy preparation procedure. The created PCM composite ensures that the temperature of the food item is maintained for a longer time throughout the meal delivery time while using less energy.

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REFERENCES

- S. Khot, N. Sane, and B. Gawali, Experimental Investigation of Phase Change Phenomena of Paraffin Wax inside a Capsule, SSRG Int. J. Eng. Trends Technol., 2(2) (2011) 67–71.
- [2] L. A. Naeem, T. A. Al-Hattab, and M. I. Abdulwahab, Study the Performance of Nano-Enhanced Phase Change Material NEPCM in Packed Bed Thermal Energy Storage System, SSRG Int. J. Eng. Trends Technol., 37(2) (2016) 72–79.
- [3] A. F. Momin and M. H. Attal, Experimental Analysis of Household Refrigerator Compatible with Phase Change Material, using R290/600a Blend as Refrigerant, SSRG Int. J. Eng. Trends Technol., 42(4) (2016)183–188.
- [4] V. Bamane and C. Papade, A Review Paper on Nano mixed Phase Change Material for Indoor and Outdoor Solar Cooker Application, SSRG Int. J. Eng. Trends Technol., 43(7) (2017) 393–397.
- [5] A. B. Umar, M. K. Gupta, and D. Buddhi, Thermal cycle testing of a few selected inorganic salts as latent heat storage materials for high-temperature thermal storage, SSRG Int. J. Eng. Trends Technol., 69(8) (2021) 17–25.
- [6] T. Amberkar and P. Mahanwar, Manufacturing Technology of Shape-Stabilized Phase Change Materials, Int. J. Res. Rev., 5(7) (2018) 24–34.
- [7] T. Amberkar and P. Mahanwar, Review on thermal energy storing phase change material-polymer composites in packaging applications, Mater. Proc., 7(1) (2021) 14.
- [8] F. Arjona, T. Calvet, V. Métivaud, and D. Mondieig, Application of the N-Alkane molecular alloys to thermally protected containers for catering, Boletín la Soc. Española Cerámica y Vidr., 39(4) (2000) 548–551.

- [9] R. Booska, Thermal receptacle with phase change material, U.S. Patent 10 595 654, (2020).
- [10] M. Agostini, Technical plate, WO 2012131471,(2012).
 [11] P. Espeau, D. Mondieig, Y. Haget, and M. A. Cuevas-Diarte, 'Active' package for thermal protection of food products, Package. Technol. Sci., 10(5) (1997) 253-260.
- [12] T. Amberkar and P. Mahanwar, Synthesis and study of microcapsules with beeswax core and phenol-formaldehyde shell using the Taguchi method, Mater. Proc., 7(1) (2021) 1.
- [13] S. P. Singh, G. Burgess, and J. Singh, Performance comparison of

thermally insulated packaging boxes, bags and refrigerants for single-parcel shipments, Package. Technol. Sci., 21(1) (2008) 25-35.

- [14] M. E. Hossain, M. I. Khan, C. Ketata, and M. R. Islam, Comparative pathway analysis of paraffin wax and beeswax for industrial applications, J. Charact. Dev. Novel Mater., 1(4) (2010) 1-13.
- [15] S. Naderizadeh et al., Superhydrophobic Coatings from Beeswaxin-Water Emulsions with Latent Heat Storage Capability, Adv. Mater. Interfaces, 6(5) (2019) 1-11.