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Preparation and Characterization of Flexible Phase Change Material for Compact Electrical Applications

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Abstract

The development of advances in digital technologies, the compact electronic devices plays an important role but the space constraints and irregular surfaces in the device makes it hard to dissipate heat with the help of external medium. They overheat in most of the cases and become a common cause for failure of the device. Phase Change Materials (PCM) release or absorb heat at its melting temperature to its better thermal management. But the PCM have limitations as its brittleness could not occupy the irregular surfaces of compact electronic devices and undergo deformations during operation, a new class of material Flexible PCM (FPCM) developed to endure a certain amount of deformation and make compact contact with irregular surfaces of objects, making them ideal for many smart applications. In this work, the newer thin film FPCM are prepared using Olefin Block Copolymer and paraffin wax at different combinations on mass basis and field emission SEM and XRD characterization of samples are studied and reported. This paper presents selection of optimal PCM and copolymer, preparation of FPCM and detailed analysis of the sample report and electrical device applications. This report provides a basis for implementing and optimizing phase change thermal management techniques in electrical devices. The experimental results shows PCM 45 and 55 wt% co-polymer combination of FPCM having better thermal management in Electrical Applications. Thermal analysis will be studied in future.

Keywords: Flexible phase change materials, Compactness, Miniaturization, Heat transfer, Thermal management

Introduction

The growth of the electronic industry has a key role in the digitalization of the economy. Electronic industry progress is on newer designs with more compactness and miniaturization. Thermal management in these new product designs is more important because each new device would squeeze more power and performance in ever-smaller packages. The new designs would be of no use because poor thermal management would degrade their performance, as the performance, lifespan and durability of electronics are highly dependent on the working temperature. The more worrying part is, although the electronic device size has been reduced the traditional cooling techniques increase the overall size [1, 2]. The forced convection cooling with a fan necessitates the use of large, cumbersome equipment. Furthermore, due to noise, maintenance and other factors, fans are unsuitable for use with portal and compact devices with increased power consumption [3].

An alternative to the existing cooling systems, PCMs are gaining attention due to the temperature stability, high latent heat of fusion, high energy density and high specific heat. During melting, heat is absorbed and released into the atmosphere during the freezing process. Because of the small volume changes involved, developers are more interested in solid-liquid conversions. During the transition phase, the PCM controls the component's temperature rise for a length of time known as the effective thermal control time. It assures that PCMs can help in the thermal management of electronic devices. The average operating temperature of various electronic chips is between 50 to 120 °C. Anything above would be called a case of overheating. Furthermore, humans are capable of tolerating temperatures of up to 49 °C (even that would be uncomfortable). They are included in the thermal control design of portable electronic devices as such basic criteria.

To get over this problem, the polymer stabilized flexible phase change materials are under investigation. Hawlader *et al.* [12] used a coacervation process to make shape-stabilized PCMs and discovered that a coating with a high concentration of HCHO (formaldehyde) is particularly effective in preventing liquid leakage. Fok used n-eicosane with finned heat, and looked at the device's power and orientation that have an effect on the thermal control performance. Liu *et al.* [15], utilized in situ dispersion to disperse paraffin into an inorganic silica gel polymer, well microencapsulated PCMs were produced using the greatest proportion of paraffin −69.12 wt%. The leakage problem, thermal contact resistance and inadequate installation between PCM and controlled devices, on the other hand, have not been adequately handled, preventing their use in real energy storage and thermal control engineering. Fortunately, an olefin block copolymer (OBC) has the qualities that are required. Dow Chemical Company created it using a phase morphology that consists of crystallizable blocks and an amorphous dispersion phase. Hustad *et al.* [16]; Kamdar *et al.* [17], studied the properties of OBC, which suggests that it can be used to form a FPCM.

In this experimental study, organic phase change materials are selected as base material by keeping in mind the non-corrosive and non-toxic properties, along with good thermal stability of organic PCMs. Two different types of FPCMs, paraffin wax and palmitic acid as PCM blended with OBC. The limitations are concerned; the work is carried out on LED bulb, which has nearly the same working temperature range of other electronic chips. Two distinct thermal management systems configurations are taken into consideration. In the first case, an LED bulb is connected to the power supply; its plate and casing temperatures are noted with increasing time. In the second case, the different FPCM sheets that we prepared are carefully placed on the metal plate surrounding the LEDs and then the temperatures are noted. A comparison is then drawn between the FPCM films with different compositions and components.

Materials and methods

Material selection of phase change materials

The PCM used in the experiment is taken after considering the properties required such as high latent heat, high thermal/chemical stability and economical. Keeping in mind the above properties, Paraffin Wax and Palmitic acid having latent heats −253 and 166.3 J/g are selected and Industrial grade paraffin and palmitic acid were used in the experiment.

Selection of polymeric material

PCM used is brittle in nature, it difficult to installed on irregular surfaces of electronic devices, so in order to impart flexibility and surface covering property into our PCM we used the polymeric compound, Olefin Block Copolymer (OBC), as a supporting material having excellent elasticity, toughness, low temperature ductility and low shrinkage, with a density of 0.870 g/cm^3 and melt flow rate of 5.0 g/10 min (2.16 kg, 190 °C) [16, 17].

Preparation

After selecting the PCM and Polymeric compound, different samples are prepared by varying the composition of PCM and Copolymer as shown in **Table 1**. Initiating with 15 wt% PCM and 85 wt% polymeric compound and further increasing the wt% of PCM by 15 up to 60 %.

Table 1 wt% composition and wt in g of samples FPCM.

S. No.	PCM (wt %)	PCM (wt in g)	Co-polymer (wt in g)	Co-polymer (wt %)
1	15	1.8	10.2	85
2	30	3.6	8.4	70
3	45	5.4	6.6	55
4	60	7.2	4.8	40

Samples are made with constrained as total weight to be 12 g i.e. weight of PCM + weight of copolymer. Copolymer is melted in a bath with constant stirring. Then PCM was added (which was already melted separately with use of the Heating Mantle) into the bath and stirred both thoroughly until it mixed properly.

By applying this procedure, 8 thin films were required to be made, out of which 4 were of Paraffin Wax & Olefin Block Copolymer (OBC) and the rest four were of Palmitic Acid & Olefin Block Copolymer (OBC). To get thin films of PCM a machine which could apply uniform pressure, maintain a certain temperature more than ambient and roll down the hot poured material into thin films was required. An automated lamination machine was used as shown in **Figure 1**. The hot mixture of OBC and PCM was between thick plastic sheets and then rolled down multiple times to get the desired thickness of FPCM. By repeating this process, a total of 8 thin circular films were prepared from 8 different compositions.

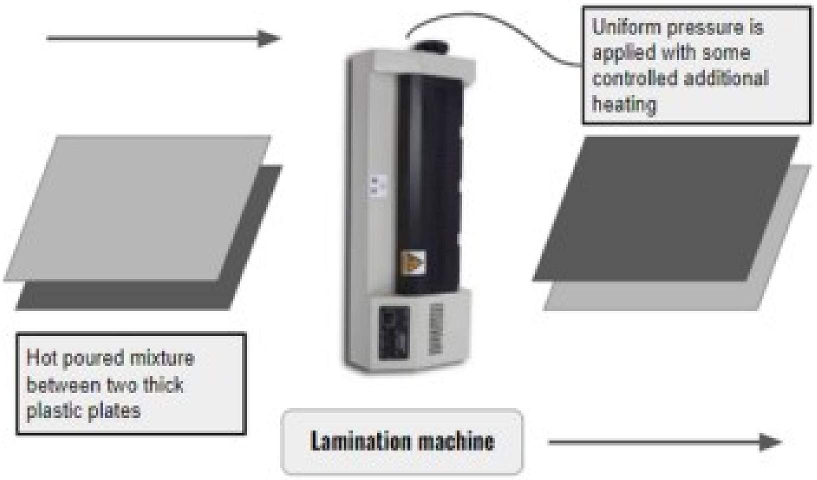

Figure 1 Schematic diagram FPCM lamination machine.

Different compositions behaved differently to the rolling process and the properties-ductility, flowability, thermal stability and surface texture varied for different compositions. The samples of FPCM (paraffin + OBC) and their microscopic images are shown in **Figure 2**.

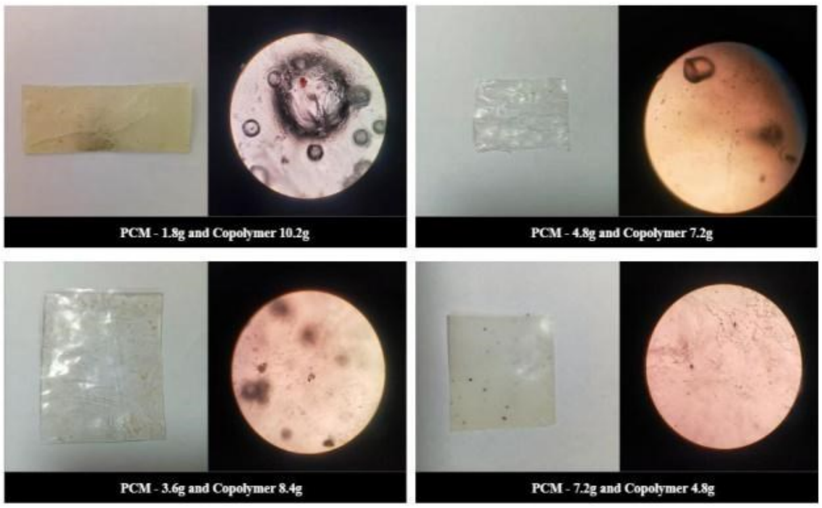

Figure 2 Samples of FPCM (paraffin + OBC) and their microscopic images.

The above mentioned compositions of PCM could not yield FPCM in case of Palmitic Acid, so lower compositions of palmitic acid - (8, 10, 15 and 30 wt %) were used and then FPCM was formed as shown in **Figure 3**.

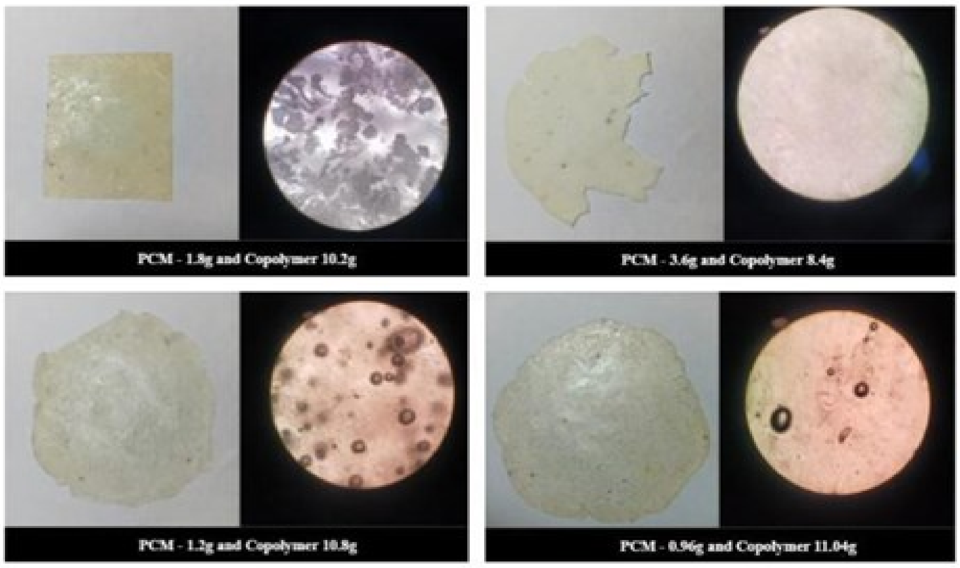

Figure 3 Samples of FPCM (Palmitic Acid + OBC) and their microscopic images.

Experimental setup

LED bulb application

In the past few years, the usage of LED bulbs has increased rapidly all over the world because of its technology, energy saving, economical, being more compact it has spread widely. LED bulbs consist of different electrical and electronics components, certainly producing heat non-uniformly through the non-planar surface which results in overheating, which has become the main factor of non-functioning LED bulbs. If this extra heat generated can get dissipated out of the bulb, then it is expected that the problem of non-functioning of the bulb due to overheating is solved. It's working temperature range between 40 to 80 °C.

Specification of LED bulb

Brand: ECOLINK

Spec: 12 W (220 – 240 V – 50 Hz)

Experimental procedure

For the selected application, the FPCM thin film must be in the shape of annulus, so that it can be applied on the top surface of the metallic plate of the LED bulb and cover the entire circular surface with hole at the central axis. FPCM film applied on the annular metallic plate of LED is shown in **Figure 4**.

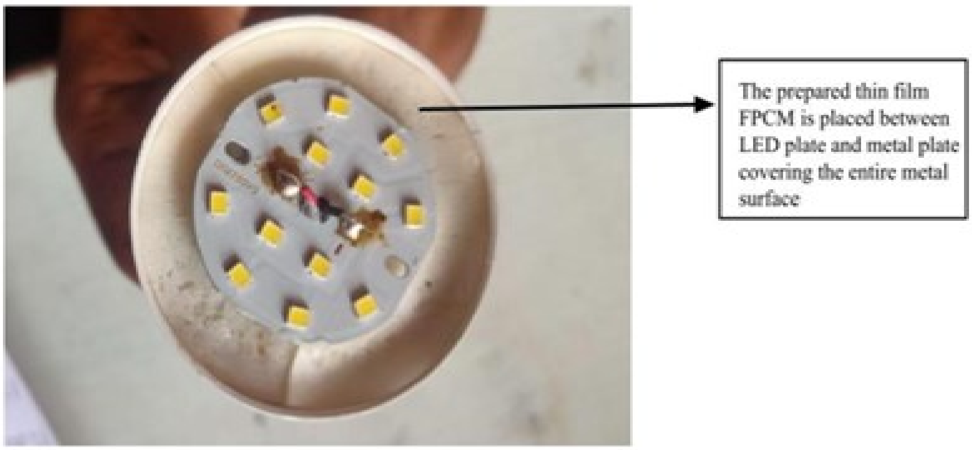

Figure 4 FPCM film applied on the annular metallic plate of LED.

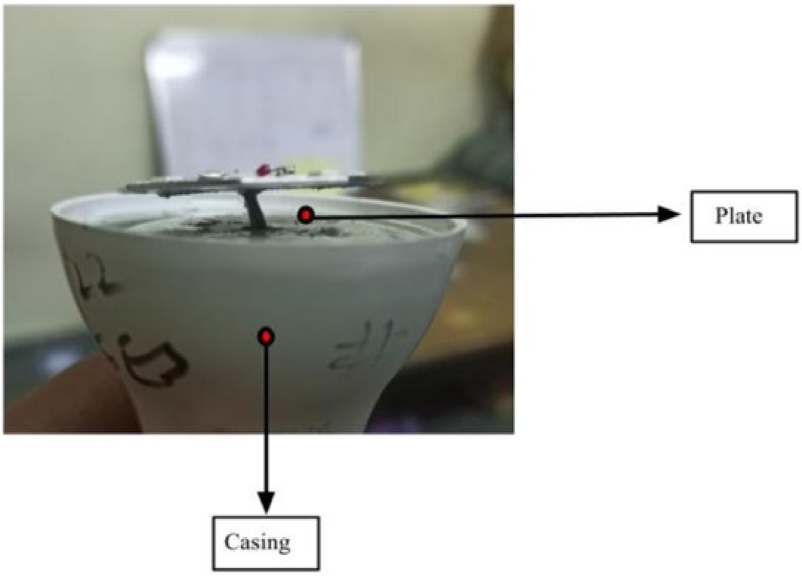

Figure 5 Plate and casing of a LED bulb under consideration.

Figure 5 shows the casing and metallic plate of the LED bulb. The circular FPCM film was cut from the middle of the annular FPCM thin film was used in a LED and the temperature at casing and plate of the bulb was recorded by using a temperature gun (used both IR for casing and thermocouple for plate).

The FPCM film was carefully placed on metallic plate without removing LED plate and the wiring. The LED bulb has a metal surface diameter of 50 mm, so equal diameter film has been used to cover the entire surface such that the entire heat dissipating out from the metal plate will get in contact with the applied film. Even the outer covering of the LED was reinstalled after placing the FPCM film inside. The thermocouple wire was kept in contact with the plate through a small hole in the lower casing of the LED. The LED was switched 'ON' at actual working conditions 12 W (220 - 240 V - 50 Hz). Total time of study = 100 min and gap between consecutive reading = 5 min. Before applying the thin film FPCM on the bulb, the temperature of plate and casing was recorded for the same time of study to see how the temperature of the casing of the bulb and plate of the bulb is changing. Later, temperature was measured after applying the FPCM film. The case temperature throughout the study was measured by the means of IR mode with the help of the temperature gun and the plate temperature was measured by the thermocouple of the temperature gun. While measuring the plate temperature throughout the study the hemispherical cap of the bulb was in its actual position and in actual condition the temperatures were measured. The same procedure was applied to measure the temperatures in case when FPCM film is applied and then temperature data was recorded for all the samples discussed.

Results and discussion

The data acquired from the experiment technique resulted in a variety of graphs from which inferences might be drawn. The composition with 15 and 60 wt% Paraffin could not yield the desired results. The 45 and 30 wt% composition obtained favorable results. The graphs are plotted using the curve fitting tools in the MATLAB software. **Figure 6** shows the casing and metallic plate of the LED bulb without FPCM, the temperature at casing and plate of the bulb was recorded by using a temperature gun (used both IR for casing and thermocouple for plate). Plate temperature reaches the maximum values to 75.1 °C after 80 min and casing temperature reaches to maximum value of 64.1 °C after 80 min. For the first 5 min, the temperature rise was more for casing, but as the working time increased, the temperature of the plate began to climb significantly.

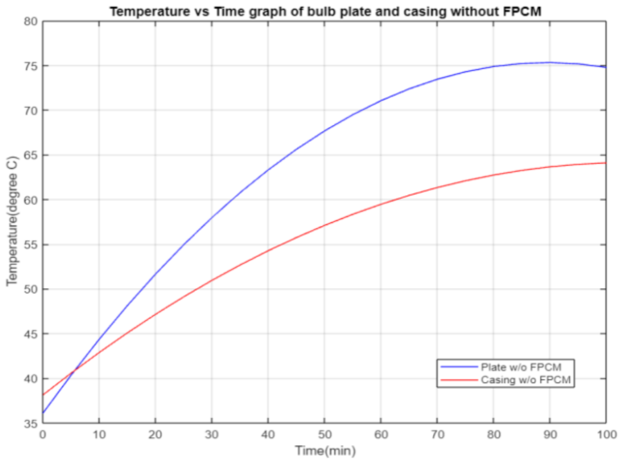

Figure 6 Comparison of temperature variation of plate and casing without FPCM.

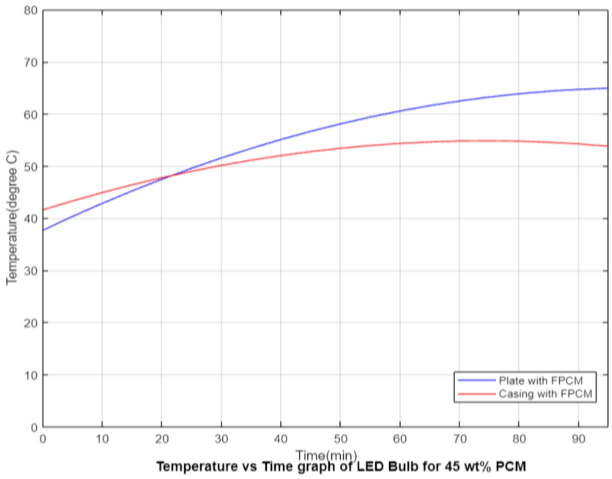

Figure 7 Comparison of temperature variation of plate and casing with 45 wt% FPCM.

The casing is in constant touch with the ambient conditions, it has initially lower temperature but as the melting temperature of the PCM is reached, the heat from plate is absorbed but the casing receives extra heat through radiation mode. **Figure 7** shows the plate and casing temperature with respect to time for 45 wt% of paraffin wax, casing temperature reaches to 55 °C at 80 min and 65 °C for bulb plate temperature when at 95 min.

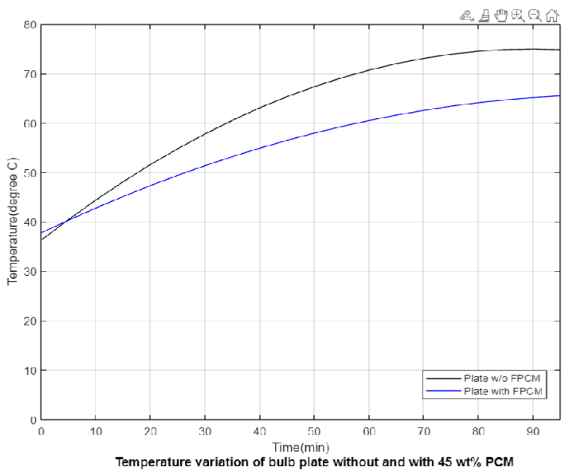

Figure 8 Comparison of temperature variation of plate without FPCM and with 45 wt% FPCM.

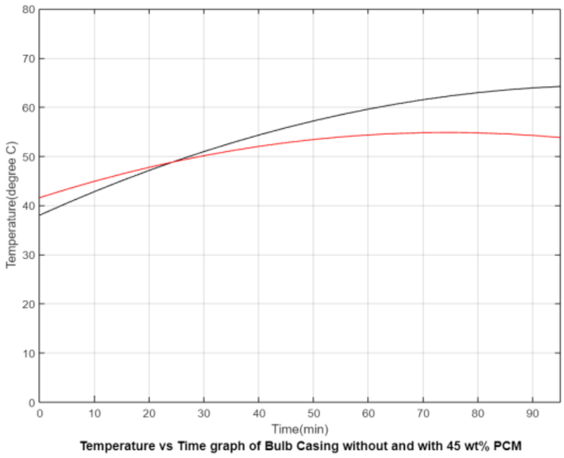

Figure 9 Comparison of temperature variation of Bulb Casing without FPCM and with 45 wt% FPCM.

Figures 8 and **9** shows the significant temperature drops between bulb casing with and without 45 %wt paraffin wax values of 10 °C was observed in the plotted graph. This shows that the FPCM film was essentially working well in the actual conditions.

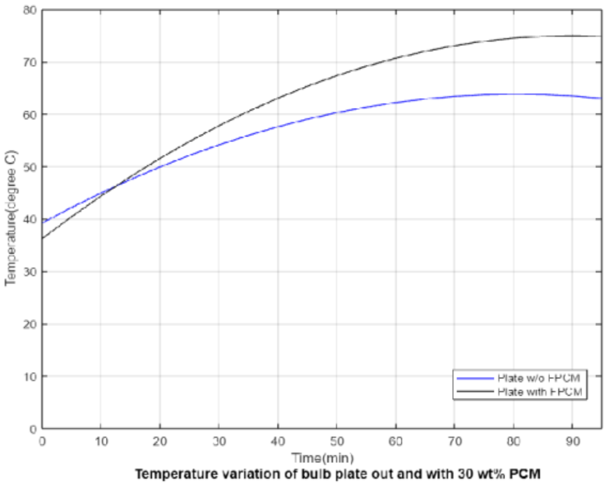

Figure 10 Comparison of temperature variation of Bulb Plate without FPCM and with 30 wt% FPCM.

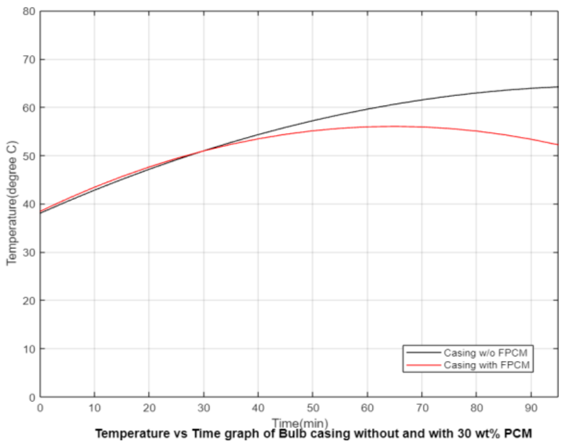

Figure 11 Comparison of temperature variation of Bulb Casing without FPCM and with 30 wt% FPCM.

Figure 10 shows variation of temperature of LED bulb plate with time having the FPCM with and without 30 wt% paraffin obtained the maximum value of 62 °C maintains from 80 min. Bulb plate temperature without FPCM reached to 75 °C at 90 min.

Figure 11 shows variation of temperature of casing with time having the FPCM with and without 30 wt% paraffin obtained the maximum value of 56 °C maintains from 60 to 70 min. Casing temperature without FPCM reached to 64 °C at 95 min.

The tests were carried with Palmitic acid as a PCM but it showed very poor form stability. It was not thermally stable as well. Only one 15 wt% composition could form stable FPCM, which had high leaking problems with increasing temperature.

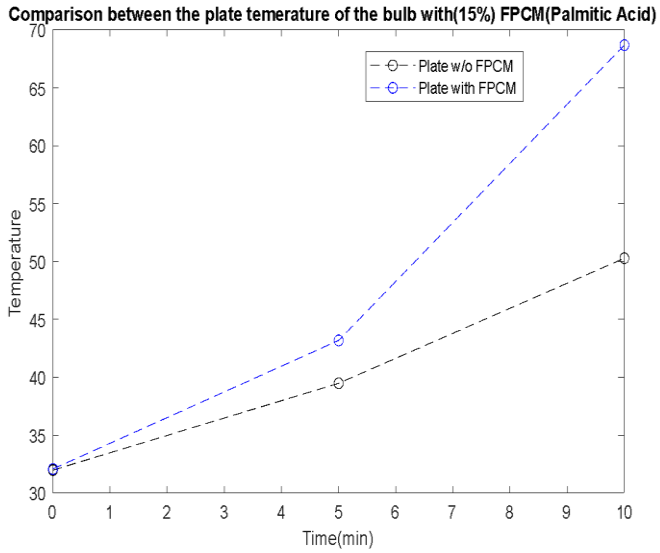

Figure 12 Comparison of temperature variation of bulb plate without FPCM and with 15 wt% FPCM.

The leaking problem of this FPCM could impair the working conditions and life of the electronic component. There were a number of other issues with employing palmitic acid as a PCM, including its toxicity, which made preparation difficult.

FPCM characterization study

X-ray diffraction

The most popular approach for determining the crystalline structure of coatings is X-ray diffraction (XRD). It contains information about the crystal orientation, phases present, lattice parameters, crystal structure, crystalline and so on. A Rigaku XRD analyzer was used to record the XRD patterns of the base material at CRFC Lab NIT Srinagar's Smart Lab X-ray diffract meter.

The X-Ray diffraction pattern of the base material (AMS 5898) was measured in steps of 0.02 ° from 5 to 90 °. Cu-K beta radiation at 40 kV, 30 mA, in continuous scanning mode with D/teX Ultra 250 was used to create the X-rays, which were measured with Cu-K beta radiation at 40 kV, 30 mA. The pattern of the X-ray diffract gram was compared to the ICDD (PDF-2 Release 2018 RDB). In powder X-ray diffraction, the diffraction pattern is obtained from a powder of the material rather than an individual crystal. Because powder diffraction does not need the formation of individual crystals, it is often easier and more convenient than single crystal diffraction. There were 2 samples for which XRD was performed, one with 30 wt% PCM (paraffin wax) and 70 wt% OBC (sample was named as P104) and another with 45 wt% PCM (paraffin wax) and 55 wt% OBC (Sample was named as P103). The diffraction pattern obtained for samples P103 and P104 are shown in **Figures 13** and **14**.

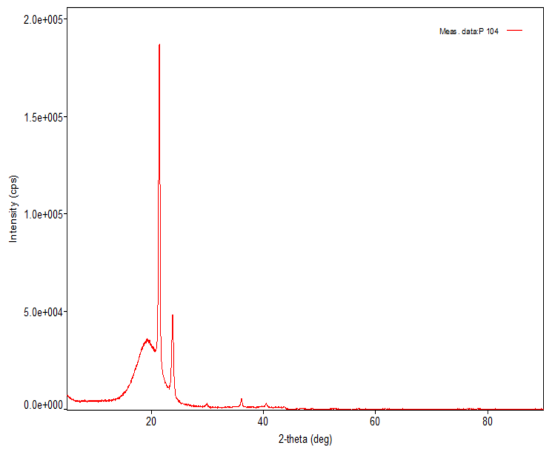

Figure 13 Diffraction pattern for 30 wt% Paraffin FPCM.

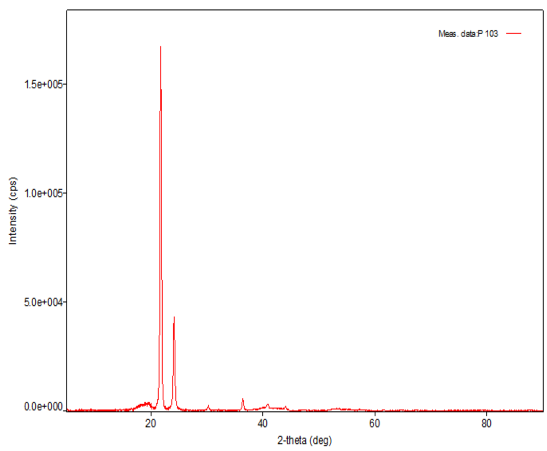

Figure 14 Diffraction pattern for 45 wt% Paraffin FPCM.

Figures 13 and **14** give a clear understanding about the nature of our samples. The diffraction pattern of amorphous materials shows humps as they do not have a periodic array with long range order whereas crystalline material shows sharp peaks. **Figure 12** has sharp peaks but **Figure 11** has hump which reconfirms that there is more quantity of PCM in P103 as Paraffin is more crystalline. From the test performed the result obtained for crystalline are shown in **Table 2**.

Table 2 Crystalline values for samples P103 and P104.

Data set name	Crystallinity (%)
P103	99.50
P104	99.99

The test results show that P104 with 30 wt% Paraffin has slightly more crystalline and more crystalline means more brittleness, hardness, density and more intermolecular interactions at higher temperatures. But, the intended function of the material to be developed requires good elasticity, toughness and stability at higher temperatures.

FIELD emission scanning electron microscopy

The FESEM (Field Emission Scanning Electron Microscope) is a useful tool for determining the morphology and structure of surfaces. The FESEM features a substantially brighter electron source and a narrower electron beam than a traditional SEM, allowing observation and imaging magnifications of up to 500 Kx. Using a ZEISS Gemini SEM 500, FESEM pictures of the substrate material were taken at the CRFC Lab, NIT Srinagar. Throughout the operation, the vacuum in the system was kept at or below the specified level of 1×10^{-5} mbar.

The surface morphology of the PCM was captured using FESEM shown in **Figure 15**. The surface morphology of the FPCM was captured using FESEM for varied weight percentages (30 and 45 %) of Paraffin. It is obvious from the **Figures 16** and **17** that the surface texture of the latter 2 is smoother than the former 2. When the microscopic image from the FESEM test was examined closely, there was no significant immiscibility between PCM and OBC, indicating that the OBC network had a high potential for uniformly dispersing PCM. Under high magnification, **Figures 18** and **19** exhibits a dense and smooth surface.

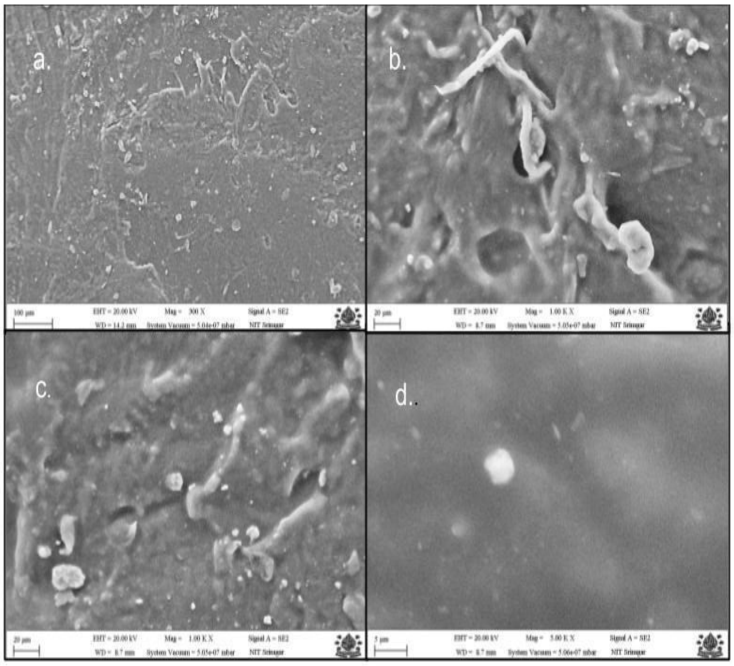

Figure 15 FESEM images of pure PCM at a) 300X, b) 500X, c) 1,000X, and d) 5,000X.

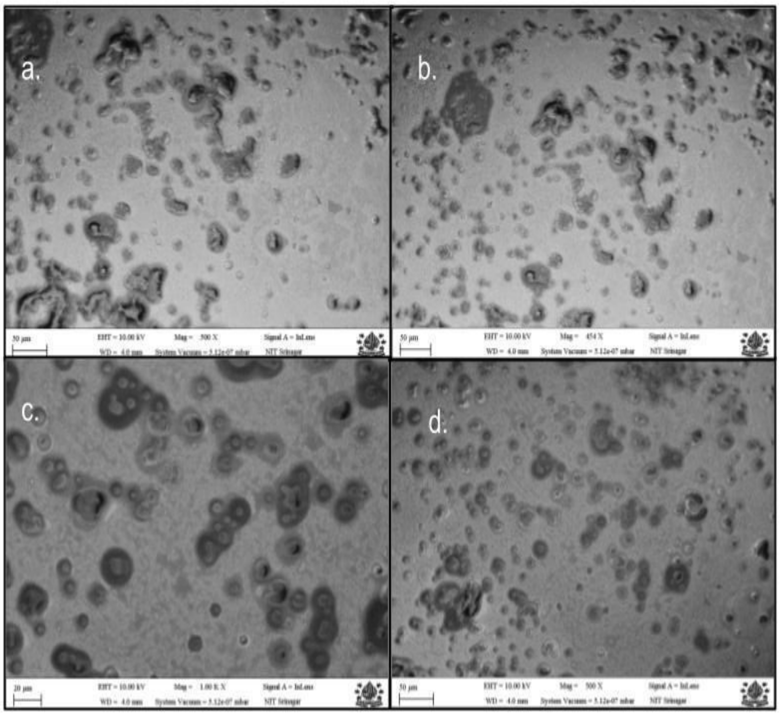

Figure 16 (a) FESEM images of OBC at a) 500X, b) 454X, c) 1,000X, and d) 500X.

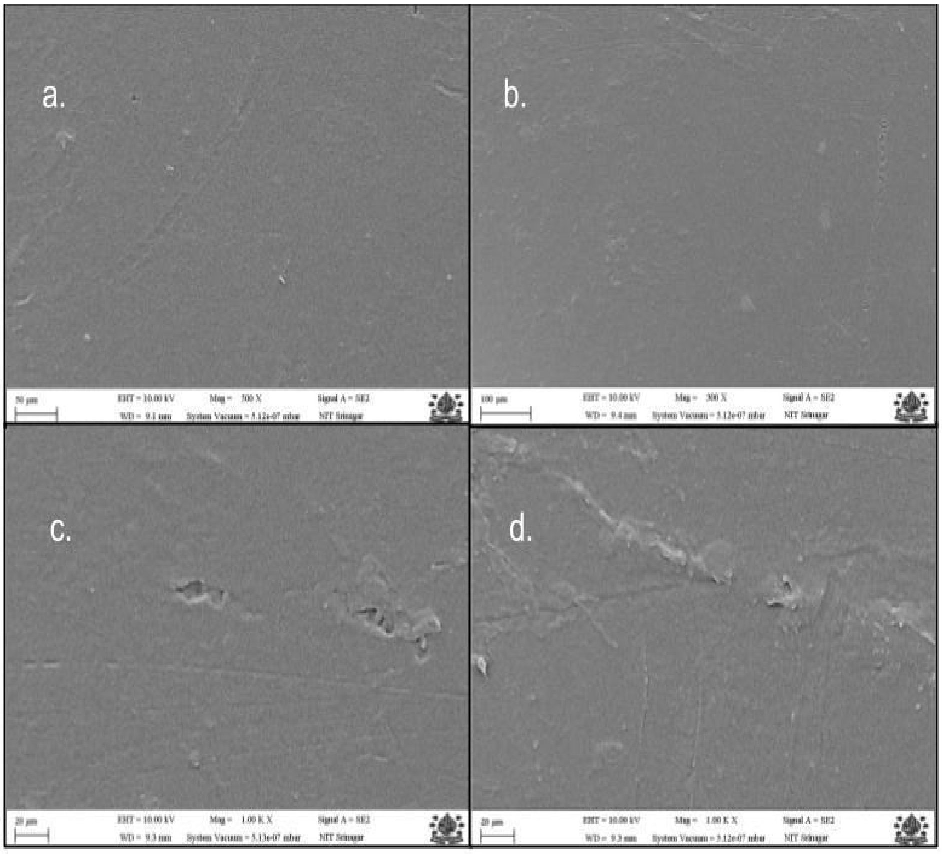

Figure 17 FESEM images of 45 wt% FPCM at a) 500X, b) 300X, c) 1,000X and, d) 1,000X at different system vacuum.

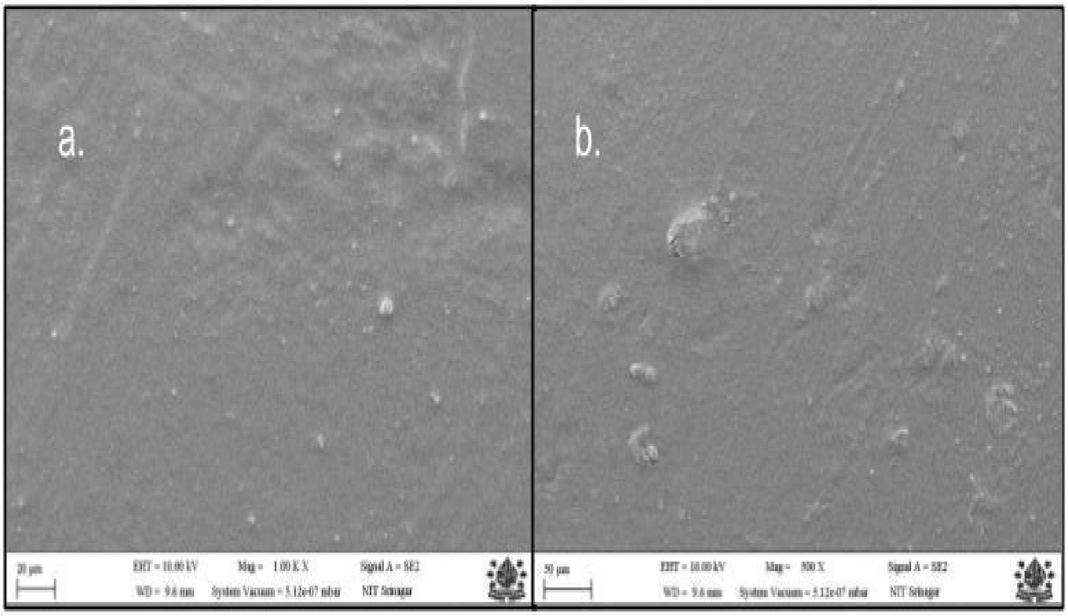

Figure 18 FESEM images of 30 wt% FPCM at a) 1,000X and b) 500X.

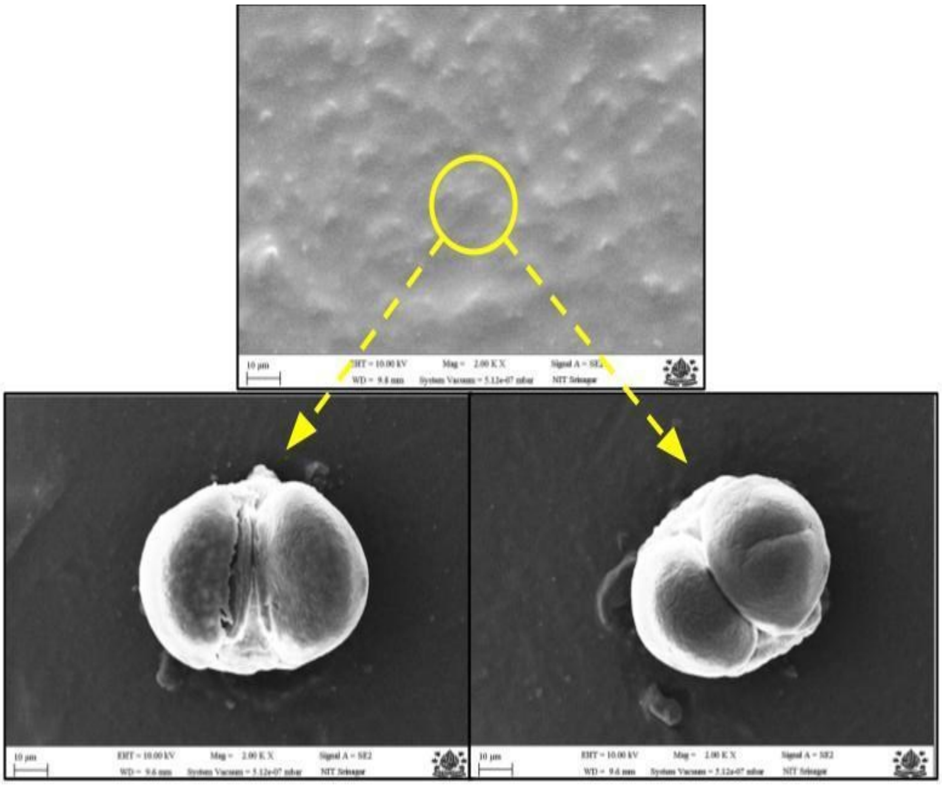

Figure 19 FESEM images of 30 wt% FPCM at 2,000X under the Working Distance (WD) of 9.6 and 9.8 mm.

Research improvement

The developed FPCM used for other electronic cooling, thermoelectric generator heat sink and solar photovoltaic module cooling applications.

Conclusions

High latent heat PCM's has made it one of the most promising materials for a variety of thermal management applications. PCM's utility is restricted, however, due to leakage, low heat conductivity value and high stiffness. The easier production of a novel form-stable and thermally induced flexible paraffin-olefin block copolymer composite PCM was developed and its characterization were studied for different samples on at different weight basis in this paper. Palmitic acid was considered as a PCM at first, the FPCM created with it was extremely brittle and unstable when larger amounts were applied. The Paraffin-OBC FPCM, which contains 30 wt% Paraffin, has a higher heat absorption capacity and lower leakage. The FPCM film with 45 wt% paraffin, on the other hand, has a low crystalline, which means it has greater elasticity, hardness and brittleness. **Table 3** shows summary of LED plate and casing temperature for different PCM %wt.

Table 3 Summary of LED plate and casing temperature.

S. No.	PCM %wt	Chasing temperature (°C)	Bulb plate temperature (°C)
1	15	64	77
2	30	62	75
3	45	54	65
4	60	58	69

The FPCM layer greatly reduces the temperature of the casing as well as the plate, according to the LED bulb testing. The temperature drop in the case is greater than the temperature drop in the plate because the case was in direct contact with the outer surroundings. Finally, the as-prepared composite PCM *i.e.* FPCM has excellent heat storage capability, as well as strong thermal, chemical stability along with the forming stability. The paraffin phase change temperature could be altered to suit various application conditions. Furthermore, the thermally induced flexibility aids in the reduction of thermal contact resistance and the enhancement of installation. The XRD and FESEM results revealed that the components of the composites were physiochemically compatible. Thermal gravity analysis and digital scanning calorimetry properties will be studied in further.

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