Journal of Engineering Science and Technology
Special Issue on AIEC2024 Vol. 20, No. 1 (2025) 14 - 25


# HARNESSING COCONUT SHELL CARBON MICRO PARTICLES FOR ENHANCED OPTICAL ABSORBANCE AND SUSTAINABLE ENERGY STORAGE IN ORGANIC PHASE CHANGE MATERIALS

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**Abstract**

Initiation of heat energy absorb, and release depends on the surrounding temperature and phase transition temperature of PCM. Nevertheless, the commonly hindered practical issue of PCMs is poor optical absorbance, fluctuation in energy storage and low thermal conductance. The current research aimed to assess the chemical constancy, improve the optical absorbance, decrease the optical transmittance & to analyse the variation in melting enthalpy of organic PCM functioning at 50 °C via dispersion of coconut shell biochar-based carbon micro particles (CSCMPs). The synthesized CSCMPs are dispersed within the PCM matrix at weight fraction of 0.2%, 0.4%, 0.6%, 0.8% and 1.0% via melting-blending-sonication process. The developed composite organic PCMs are analysed with a series of material characterization experiments by employing Fourier Transform Infra-Red spectroscopy (FTIR), Ultraviolet visible spectroscopy (UV-Vis), and differential scanning calorimeter (DSC). Results ensure, chemical stability, improved optical absorbance by 63.6%, decreased optical transmissibility by 64.3%; and surge in energy storage ability from 158.2 J/g to 190.1 J/g. The findings also provide valuables information towards design and improvement of composite PCM materials for various thermal regulation application from buildings to electronic devices.

Keywords: Chemical stability, Coconut shell biochar, Optical absorbance, Phase change material, Thermal energy storage.

## 1. Introduction

In recent times, there has been a notable focus on organic phase change materials (PCMs) attributed by their exceptional capability to store and release thermal energy during phase changeovers [1]. These materials offer great potential for a wide range of applications, such as thermal energy storage (TES), passive solar heating [2], battery thermal management [3] and temperature regulation in buildings [4]. Nevertheless, the practical implementation of organic PCMs is frequently impeded by limitations in optical absorbance and energy storage efficiency [5]. It is imperative to tackle these obstacles to enhance the performance and practicality of systems based on organic PCMs. The effective capture and utilization of solar radiation heavily rely on the optical absorbance of phase change material (PCM) substances. PCM-based systems can efficiently harness solar energy for heating or cooling purposes by enhancing their optical absorbance. This, in turn, reduces the dependence on conventional energy sources and decreases carbon emissions [6]. Furthermore, it is crucial to optimize the energy storage capacity of organic PCMs to maximize thermal energy storage and enhance overall system performance.

To enhance energy efficiency and comfort in buildings, the installation of PCMs for thermal regulation is a widely opted technique which involves series of steps. The first step is to assess the building's thermal performance requirements by taking into consideration factors such as local climate, building orientation, and existing insulation [7]. Next PCMs are selected based on their phase change temperatures, which should align with the building's cooling or heating needs. PCMs can be integrated by embedding them into building materials like walls, ceilings, or floors, or by using PCM-infused panels and drywall [8]. After installation, continuous monitoring of PCM performance is essential to ensure they are effectively managing indoor temperatures and achieving the desired energy savings. Properly installed PCMs can lead to significant reductions in heating and cooling costs while improving overall indoor comfort. Figure 1 presents the schematic of heat transfer between building and PCM for effective thermal regulation.

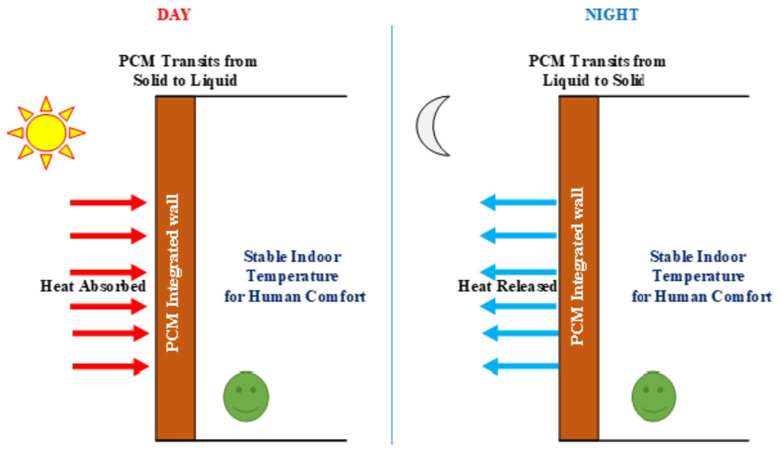


**Fig. 1. Heat transfer in buildings with installation of PCM.**

Few common issues with organic PCM are their inability to transmit thermal energy in a quick pace, as well organic PCMs are flammable in nature.

Additionally, with long time operation, the PCM at liquid state, tend to cause leakage issue, which is significantly reduced via encapsulated PCM.

Recently, significant investigation efforts have been dedicated to developing innovative techniques that enhance the optical absorbance and energy storage properties of organic PCM materials. The inclusion of carbon-based additives like graphene, carbon nanotubes, and carbon fibres has shown promising outcomes in improving thermal conductivity and energy storage efficiency [9]. However, the high cost, complex synthesis methods, and potential environmental concerns associated with these additives have prompted the exploration of alternative materials that offer comparable or superior performance [10]. Few existing research works on biochar dispersed organic PCMs are reviewed to illustrate the research opening. Utilizing garlic stem, Xiong et al. [11] successfully synthesized biochar nanoparticles through an environmentally friendly approach. These nanoparticles were then dispersed within paraffin at different weight fraction between 1-5 wt. %. The garlic stem nanoparticles, characterized by their two-dimensional flake structure, contributed to the formation of thermally conductive links within the paraffin PCM matrix.

Wan et al. [12] conducted an experiment where they developed a form stable PCM by combining palmitic acid & biochar of pinecone in various mass ratios. These ratios included 4:7, 4:6, 5:5, 6:4, & 6.5:3.5. The study revealed that the thermal conductivity of the palmitic acid: biochar of pinecone mixture at a ratio of 6:4 increased by approximately 43.76%. This enhancement can be attributed to the effective impregnation of palmitic acid in the pores of pinecone biochar, causing in the formation of superior thermal network channels. The fruit of Pinus resinosa was subjected to pyrolysis by Mandal et al. [13] to generate biochar with enhanced porosity and surface area. To address the problem of leakage, the researchers dispersed this biochar with dodecanoic acid, creating a shape-stabilized composite PCM. The composite PCM was formulated using a 3:1 ratio of dodecanoic acid to biochar. Likewise, Kalidasan et al. [10] and Yadav et al. [14] conducted an experimental analysis to explore the potential of agro waste-based biochar with polyethylene glycol and eutectic composition of salt hydrate PCM.

Low temperature PCMs are also actively used for thermal regulation of buildings. Integration of recycled expanded glass-based shape stabilized octadecane PCM with cement mortars has led to the development of a new class of concrete material, which effectively resolves the issue of leakage during phase transition of PCMs. This innovative solution offers an energy storage potential of 241 J/g and significantly enhances energy efficiency while reducing carbon emissions [15]. The synthesis of a double shell PCM microcapsule by Lin et al. [16] has successfully enhanced the use of organic PCM in building thermal regulation. This innovative approach not only provides flame retardancy but also reduces the risk of fire accidents, overcoming a major obstacle in utilizing organic PCM for thermal regulation. A salt hydrate nano capsule with a eutectic composite shell has been developed by Li et al. [17] for thermal regulation in buildings. This encapsulated PCM boasts excellent energy storage capabilities and is non-flammable, ensuring resistance against leakage, supercooling, and fire risks. In this regard, serious of inorganic-inorganic eutectic PCMs operating at low temperature [18] was also developed however, the issue of corrosion and phase instability paved the need for organic PCM for thermal regulation of buildings. Ethylene vinyl acetate coated hydrate salt PCM developed by Chen et al. [19] exhibited excellent resistance against flame however with encapsulation the energy storage ability dropped by 75%. An intriguing study focused on the development of a phosphorous-based PCM combined with titanium dioxide nanotube, resulting in enhanced resistance to flame and leakage [20]. Polyethylene glycol was selected as the PCM for this research. The analysis revealed that most nanomaterial used to improve the thermophysical properties of PCM are toxic and costly.

Coconut shells, which are readily available agricultural waste, present a sustainable and renewable source of carbon micro particles that can enhance the properties of organic PCMs. The utilization of carbon micro particles derived from coconut shells not only offers a cost-effective solution but also contributes to waste valorisation and green sustainability. By transforming agricultural waste into valuable materials, this approach aligns with the principles of circular economy and resource efficiency. A potential strategy to address these constraints involves integrating carbon micro particles obtained from coconut shells into organic PCM matrices. The utilization of coconut shell-based carbon micro particles (CSCMPs) presents an environmentally friendly and economical approach to enhance the optical absorbance and energy storage capabilities of organic PCMs. The exceptional characteristics of CSCMPs, such as their extensive surface area, porous structure, and remarkable thermal conductivity, position them as optimal contenders for augmenting the efficiency of organic PCM composites.

This study is focused on exploring the potential of carbon micro particles derived from coconut shells to enhance the optical absorbance and energy storage capacity of organic PCM materials intended for thermal regulation at 50 °C. A detailed experimental investigation is conducted to assess the impact of CSCMPs on the chemical stability, optical characteristics, & energy storage efficiency of organic PCM composites. Various characterization methods such as FTIR spectroscopy, UV-Vis spectroscopy and different scanning calorimeter is applied to study the structural, functional and thermal attributes of the composite materials. The results of this study have substantial inferences for the progression of sustainable and effective TES solutions. By utilizing coconut shell-based carbon micro particles, it is anticipated that organic PCM composites with enhanced optical absorbance and energy storage capabilities can be developed for a diverse range of applications, including building insulation, solar thermal systems, and thermal regulation of electronic devices.

Subsequently, this experimental investigation contributes towards sustainability by leveraging the potential of renewable energy usage as well by generating useful materials from agro waste.

## 2. Materials and Methods

Materials, synthesis of carbon-based micro particle additive, composite preparation methods and characterization techniques adopted in this experimental research work is discussed in detailed in this section.

### 2.1. Materials

Organic PCM used as energy storage material in this research work was procured from Rubitherm Technologies GmbH. The energy storage ability of the procured organic PCM (RT50) was 160 J/g at melting temperature of 15 °C, with thermal conductivity of 0.2 W/m·K as provided in the material specification by vendor. As far as the coconut shell is concern, they were extracted from agro waste to ensure eco-friendly by recycling of waste material.

## 2.2. Synthesis of coconut shell biochar-based carbon micro particles

In this investigational study, carbon micro particles derived from biochar were synthesized using coconut shells collected from agro waste. The process of synthesizing CSCMPs is illustrated in Fig. 2. To commence with, the coconut shells were obtained from agro waste material and then chopped into small pieces measuring 2.0-3.0 cm. The chopped coconut shells underwent meticulous cleaning with deionized water to guarantee top-notch quality. Following this, the cleaned coconut shells were dried outdoors in an exposed solar environment. Afterward, the dried coconut shell particles were subjected to carbonization in a tube furnace in a $N_2$ air atmosphere at a temperature of 1000 °C. Following carbonization, the particles underwent extra handling utilizing a ball mill. To minimize the particle size as effectively as feasible, a wet ball milling method was utilized for a duration of 3 hours at a speed of 400 rpm, with a resting interval of 5 minutes following every 15 minutes of ball mill process. Based on the particle analysis, the developed carbon micro particles derived from coconut shells had a size range of approximately 400-800 nm. These carbon-based micro particles from coconut shells were then chosen to enhance the thermophysical properties of organic PCM.

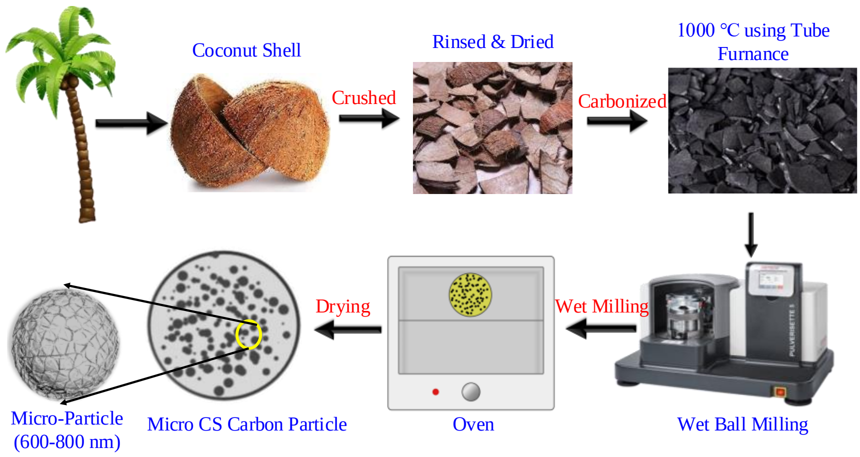


**Fig. 2. Synthesis of coconut shell-based carbon micro particles.**

## 2.3. Preparation of coconut shell micro particle dispersed composite PCM

A two-step method is employed for the preparation of the CSCMPs dispersed composite organic PCM sample, which includes melting, blending, and sonication. The process is illustrated in Fig. 3, outlining the sequential steps involved in preparing the CSCMPs dispersed PCM (RT50). Initially, 10 g of PCM is placed in a sample beaker and melted using a hot plate set at 80 °C. Subsequently, 0.02 g (0.2 wt.%) of the synthesized CSCMPs is blended to RT50 PCM at liquid state once the melting process is complete. The composite blend is then subjected to mixing using a probe ultrasonicator for 30 minutes to achieve the desired composite PCM. Adopting the same procedure, composite PCM with diverse weight fraction of

0.4%, 0.6%, 0.8% and 1.0% CSCMPs dispersed PCM were prepared. The resulting composite PCMs were subjected to further testing and characterization using sophisticated instruments to assess its chemical stability, optical properties, and thermal characteristics.

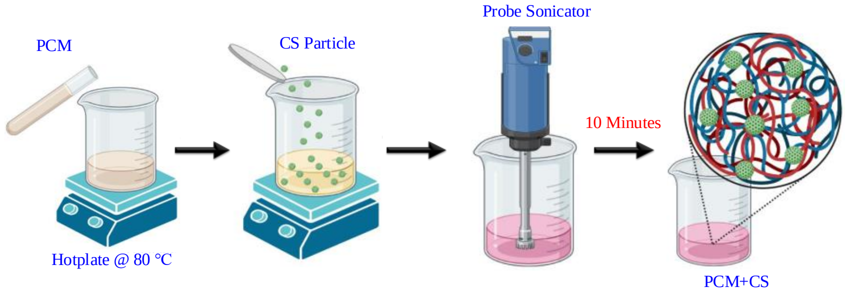


**Fig. 3. Preparation of CSCMP enhanced organic PCM.**

## 2.4. Instruments used and the characterization techniques

Various sensitive instruments were utilized to characterize the dispersed organic PCM prepared by CSCMPs. The coconut shell biochar was reduced to micro size using a planetary ball mill Model Pulverisette 5. Furthermore Table 1 provides the details of instrument, and the accuracy level used for characterization of nanocomposite.

**Table 1. Instrument details used for the experiment.**

| Equipment | Parameter measured | Accuracy | Brand |
|---|---|---|---|
| **Digital scanning calorimetry** | Latent heat | 2% | DSC 3500 Sirius of NETZSCH |
| | Melting point | 0.2 °C | |
| **Fourier Transform Infrared Spectrometer** | Functional Group | 1 $cm^{-1}$ | Perkin Elmer |
| **UV-Vis Spectrometer** | Absorbance/ Transmissivity | 0.1% | Perkin Elmer LAMBDA 750 |

## 3. Results and Discussion

The section presents the experimental characterization results of CSCMPs dispersed PCM sample. The results include the FTIR spectral curves to confirm chemical constancy, UV-Vis curves to evaluate the optical absorbance & transmittance and DSC heat flow curves to exhibit the variant in energy storage potential of the developed composite PCM.

## 3.1. Chemical stability evaluation

Chemical constancy of PCM dispersed with CSCMPs is assessed through FTIR spectroscopy to analyse their properties. By conducting FTIR investigation, the functional groups present in the samples can be determined. The FTIR spectral curve of organic PCM dispersed with CSCMPs is illustrated in Fig. 4. When exposed to infrared rays, PCM displays four IR active vibration kinds at 720 $cm^{-1}$, 1466 $cm^{-1}$, 2848 $cm^{-1}$, and 2918 $cm^{-1}$, indicating that the sample belongs to the alkane group [21]. Peak at wavenumber 720 $cm^{-1}$ exhibit the –HC = CH– functional group with rocking deformation bending; spiky spectral peak at wavenumber 1466 $cm^{-1}$ exhibit deformation bending vibration, whereas stronger sharp peak at wavenumber 2848 $cm^{-1}$ and 2918 $cm^{-1}$ display $–CH_2$ and $–CH_3$ functional group with symmetric stretching vibration respectively. Since CSCMPs do not possess a carboxyl group, they do not exhibit any IR activity and do not generate peaks in the IR spectrum. The spectral curve of all organic PCM composites, irrespective of the weight concentration of CSCMPs, closely resembles that of PCM (RT50). This similarity suggests that the CSCMPs and PCM are physically mixed, with no occurrence of chemical reactions. Moreover, this physical mixing ensures the excellent compatibility of the prepared organic composite PCM.

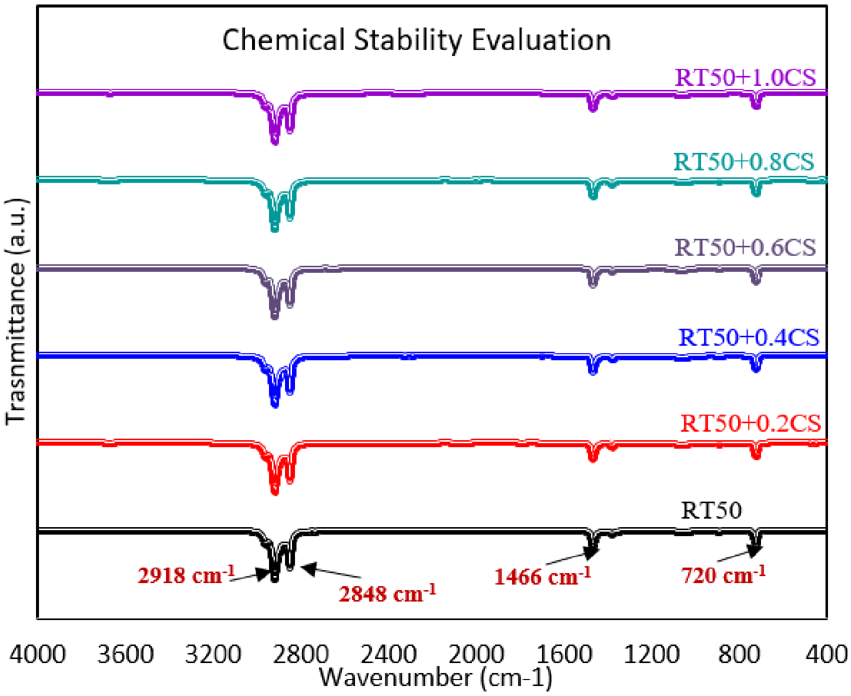


**Fig. 4. FTIR spectral curves of CSCMP enhanced organic PCM.**

## 3.2. Optical absorbance

Thermal energy storage is efficiently achieved using PCMs. To mimic the excellent solar radiation absorption capabilities of solar power, which primarily delivers thermal energy in the form of electromagnetic rays, PCMs are indispensable for energy storage. Typically, organic constituents possess transparency and high transmissibility. The absorbance and transmittance of both the base PCM and the combined PCM disseminated with CSCMPs were assessed using UV-Vis spectroscopy, and the outcomes were illustrated in Figs. 5 and 6, respectively. By passing light rays through the organic solid PCM sample using a UV-Vis spectrometer, the absorbance and transmissibility can be evaluated.

The absorbance plot in Fig. 5 clearly indicates that the CSCMPs disseminated PCM composite exhibits better absorbance compared to the base PCM. As can be inferred from the results the absorbance of PCM is 0.66 and for the composite samples with 0.2, 0.4, 0.6, 0.8 and 1.0 weight fraction of CSCMPs is 0.84, 0.93, 1.00, 1.04, 1.08 respectively. The increase absorbance in the combined PCM is attributed to the porous structure of CSCMPs that facilitates multiple reflection of the incidence radiation within the composite PCM sample as well the black coloured CSCMPs, give the composite PCM a darker texture. The extreme absorbance is witnessed for PCM+1.0%CS composite PCM (1.08), which is 63.6% greater than the base PCM (0.66). The rise in absorbance of solar radiation within composite PCM enhances the ability of PCM to be opted for solar TES.

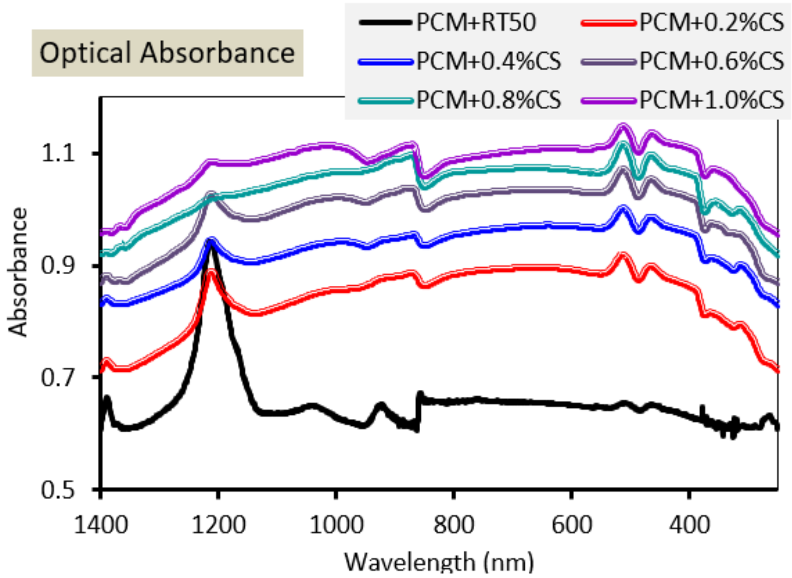


**Fig. 5. Optical Absorbance of CSCMP enhanced organic PCM.**

### 3.3. Optical transmittance

In continuation to the optical absorbance, Fig. 6 presents variation in transmissibility of the developed composite PCM samples. As low as the transmittance of PCM sample, the absorbance is expected to be higher with reflectance to be negligible. On comparison and calculation of the transmittance spectral value with the solar value, the transmissibility of PCM (RT50) is evaluated to be 22.41%. Subsequently, with the inclusion of CSCMPs at weight fractions of 0.2, 0.4, 0.6, 0.8 and 1.0 the transmissibility is calculated to be 13.7%; 11.3%; 9.7%; 8.8% and 8.0%, respectively. The decline in transmittance of the composite PCM is owing to the upsurge in better absorbance and the trapping of the incidence electromagnetic rays owing to continuous reflection and by acting opaque to the radiation. In clothing and textiles, PCM with reduced transmittance can offer enhanced comfort by better regulating body temperature. Fabrics embedded with PCM can absorb excess heat from the body when it's hot and release it when it's cold, providing a more consistent and comfortable experience for the wearer.

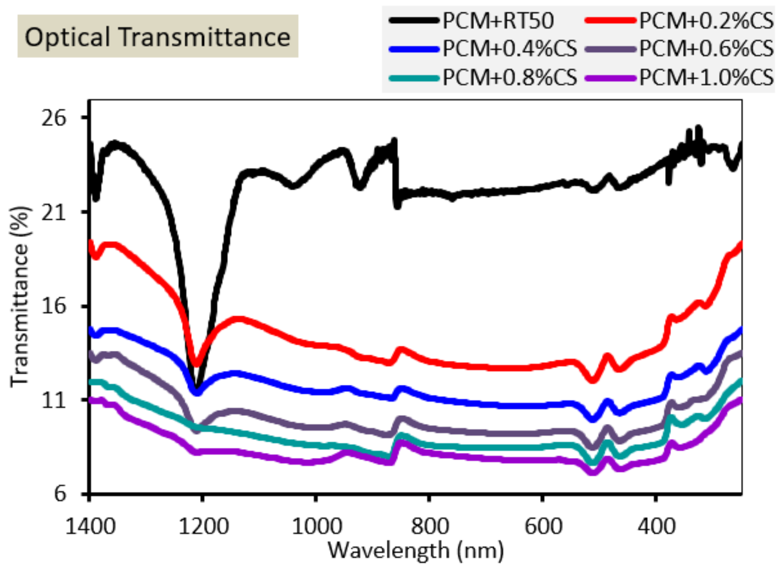


**Fig. 6. Optical Transmittance of CSCMP enhanced organic PCM.**

### 3.4. Energy storage ability

In general, PCMs are intended to facilitate energy storage and thermal regulation. Henceforth it is predominant to evaluate the deviation in energy storage ability of the developed composite PCM on dispersion of any foreign additives towards enhancing the thermal conductivity and optical property. Herewith the melting enthalpy of base PCM and its composite PCM with different weight fraction of CSCMPs are determined experimentally using DSC and the heat flow curves are depicted in Fig. 7. DSC heat flow curves provided information on the phase transition temperature and energy storage ability (melting/freezing enthalpy) during phase transition. The heat flow curve displays two peaks in the melting cycling, where the small peak indicates the solid-solid phase transition of the PCM, and the sharp peak around 50 °C indicates the solid-liquid phase transition of PCM. As mentioned in the vendor detail, PCM undergoes complete phase transition at temperature of 51°C with melting enthalpy of 158.2 J/g.

However, the phase transition temperature of the prepared sample displays an increase in solid-liquid phase transition temperature by 3-4 °C though the solid-solid phase transition temperature is almost the change. The increase in melting temperature of the composite samples is due to the addition of CSCMPs materials, with PCM. Regarding the phase transition melting enthalpy composite sample PCM+0.2%CS displays 174.3 J/g; PCM+0.4%CS displays 178.5 J/g; PCM+0.6%CS displays 189.8 J/g; PCM+0.8%CS displays 190.1 J/g and PCM+1.0%CS displays 178.5 J/g. The increase is melting enthalpy is contributed both by solid-solid transition and solid-liquid transition of PCM. In general, in most existing research work inclusion of additives to PCM tend to decrease the energy storage ability as the foreign material replace the mass of energy storage PCM material and causes reduction in melting enthalpy. Meanwhile another phenomenon is the intermolecular force of hold amongst the PCM and CSCMPs that causes a tight binding and increase the energy to break the bond, this is owing to the porous structure of coconut shell that facilitates proper binding of PCM within the porous voids. Increase in energy storage capacity of the prepared composite is a beneficial inference as the increase is about 20 J/g.

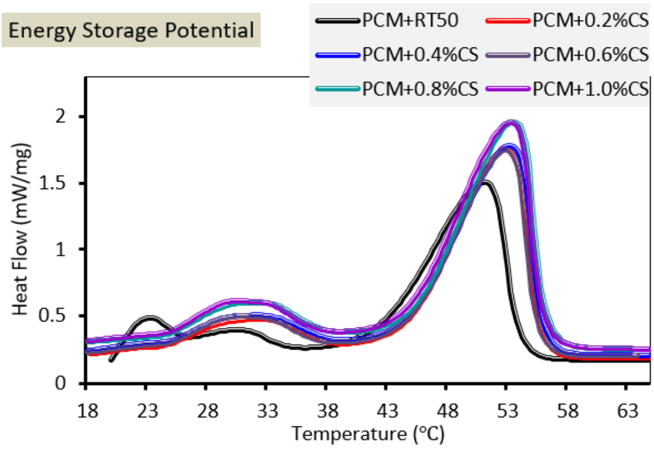


**Fig. 7. Heat Flow curves of CSCMP enhanced organic PCM.**

## 4. Conclusions

This research work involves synthesis of carbon biochar based micro particles intended to enhance the thermophysical properties of PCM. Few closing remarks from the examination are listed beneath.

- Laboratory synthesised CSCMPs are of the size 400-800 nm and exhibit greater potential to advance the optical and thermal features of organic PCM
- FTIR spectral curves ensures the chemical constancy of the developed composite PCM samples as the dispersion of CSCMPs at different weight fraction doesn't cause appearance of any new peaks.
- Optical absorbance of composite PCM increased from o.66 to 1.08 for the composite sample PCM+1.0%CS. Meanwhile the transmittance decreased from 22.4% to 8.0%, this improves their applicability with solar thermal systems.
- Phase transition temperature depicts slight variation of about 3-4 °C, whereas the melting enthalpy amplified from 158.2 J/g to 190.1 J/g for PCM+0.6%CS composite sample.
- The developed composite sample are economically cheaper and supports towards sustainable future, as CSCMPs are synthesised from agro waste, as well as PCM facilitates thermal regulation in a passive way.

**Abbreviations**

| | |
|---|---|
| CSCMP | Coconut Shell Biochar based Carbon Micro Particles |
| DSC | Differential Scanning Calorimeter |
| FTIR | Fourier Transform Infrared Spectroscopy |
| PCM | Phase Change Materials |
| TES | Thermal Energy Storage |
| UV-Vis | Ultraviolet Visible Spectroscopy |

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