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Investigations on thermal properties of MWCNT-NBN Paraffin Wax phase change material for thermal storage applications

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Abstract

The research article addresses the effect of multi-wall carbon nanotube (MWCNT) and nano-boron nitride (NBN) hybrid composite powders on thermal properties of the paraffin wax for thermal storage applications. Five different phase change material (PCM) samples were prepared with 100 paraffin wax, 99.5 paraffin wax + 0.5 MWCNT, 99.5 paraffin wax + 0.5 BN, 99 paraffin wax + 0.5 MWCT + 0.5 BN and 98 paraffin wax + 1 MWCNT + 1 BN mass percentage compositions. The size of the secondary particles MWCNT and NBN was assessed using transmission electron microscope (TEM). After PCM preparation, the morphology and distribution of the secondary particles were evaluated using field emission scanning electron microscope (FE-SEM). The phase change of MWCNT and NBN was evaluated using X-ray diffraction (XRD) technique. Differential scanning calorimetry (DSC), thermogravimetric analysis (TGA) and thermal conductivity tests were carried out on the PCMs to assess physical and thermal properties. The results revealed that hybrid nano-composite powders with paraffin wax provide better thermal conductivity of paraffin wax which increased from 0.18 to 0.31 W m^{-1} K^{-1}. However, the distribution of MWCNT and NBN extended the thermal degradation of paraffin wax and solidification temperature. Increasing the mass % of MWCNT and NBN reduced the melting point of paraffin wax from 64.70 to 62.52 °C. Further, the solidification temperature of paraffin wax increased while increasing the mass % of MWCNT and NBN from 56.01 to 60.13 °C. This research revealed that thermal properties of paraffin wax were significantly increased with the increment of mass % of composite powders (MWCNT and NBN) addition.

Keywords PCM · Characterization of paraffin wax · MWCNT · NBN and thermal conductivity

Introduction

Nowadays, storing the thermal energy is of utmost importance and many researches are focused in this area. Consumption of energy in domestic and industrial needs is more than what is produced. So scarcity of energy is increasing day by day [1–3]. Sun is the only natural source and available for limited duration and it is highly volatile. So, researchers developed thermal energy storage with phase change materials for satisfying the energy demands. In phase change materials energy is stored in the form of sensible heat, latent heat and in terms of chemical reactions subject to the nature of the material [4]. Amongst the various forms of energy storage material, PCMs are most effective because of superior energy density and capacity to store huge amount of thermal energy in the form of latent heat. On the other hand, greenhouse gas emissions are on the rise because of consumption of fossil fuel [5]. This could impact global climate change and hence creating damages to earth. However, fossil-based fuels are one of the major energy sources to fulfill the energy requirements. Presently most of the energy storage technology is environmentally unsound. Wide research was performed by various researchers on organic and inorganic phase change materials to evaluate their ability for energy storage. PCMs quickly and reversibly exchange their phase from crystalline and amorphous in micro-seconds upon heating via electrical, optical or other means. Of the many energy storage materials available researchers

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prefer paraffin wax because of properties like large energy density, low vapor pressure, better thermal stability, minimal supercooling and low cost [6, 7]. But the paraffin wax has less thermal conductivity. Researchers made many attempts on PCM material for increasing the thermal conductivity and came up with few recommendations such as imparting porous material addition, high conductive material addition in the paraffin wax. Some of them recommended addition of high conductive nano-particles too. Researchers have adopted melt blending in order to fabricate high conductive PCMs into matrices (Base PCM). Normally additives used to enhance the thermal conductivity (k) of original PCMs are metal, ceramics, polymers, carbon-based material, etc.

The main limitation of paraffin wax is low thermal conductivity which is due to interfacial thermal resistance between the PCM and reinforcing material, as well as low efficiency of charging during thermal energy storage. PCMs applications are very wide such as solar water heater and solar cooker. However, improving the thermal conductivity and enriching other properties of PCM will make them suitable for thermal energy storage systems. Generally, PCMs are reinforced with carbon and boron-based material such as MWCNT, SWCNT, B_4C and NBN. Though many carbon-based materials are there, but reinforcing the hybrid nanoparticles can be a viable solution for enhancing the thermal conductivity of the energy storage system. The addition of MWCNT and NBN lowers the interfacial thermal resistance in PCMs. For instance, hybrid nano-additives (less than 2 mass %) mass fraction creates large interfacial thermal resistance values among the nano-additives and base PCMs [8]. Moreover, increasing the mass % of nano-additives will create a network and it improves the thermal conductivity and other properties such as mechanical and thermal storage capacity. Karaipekli et al. [8] studied the thermal conductivity of PCM material by reinforcing the carbon nanotube with varying mass percentage such as 0.3, 0.5 and 1.0 and the experimental results revealed that increasing the mass percentage of carbon nanotube will increase the thermal conductivity of PCM by 11.3%. Şahan and Paksoy et al. [9] recommend that nano-zinc oxide (ZnO) with paraffin wax advances the thermal conductivity for solar thermal energy storage applications. Literature reports very few studies, where hybrid nano-additives were reinforced with paraffin wax with various mass %. The current research is focused on fabricating and studying properties of PCMs with MWCNT and NBN. The nano-particle sizes are characterized using TEM and XRD. Five different hybrid PCMs were prepared with 100% paraffin wax, 99.5% paraffin wax + 0.5% MWCNT, 99.5% paraffin wax + 0.5% NBN, 99% paraffin wax + 0.5% MWCT + 0.5% NBN and 98% paraffin wax + 1% MWCNT + 1% NBN by mass compositions. After fabrication of hybrid PCMs, the dispersion of the nano-additives was characterized using FE-SEM. The physical, thermal and thermal cyclic behavior of the fabricated PCMs is assessed using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). The obtained results are evaluated and compared with pure paraffin wax.

Experimental

Materials

Commercially available paraffin wax was used as PCM in this experiment because it was economical and delivered anticipated thermophysical properties. The paraffin wax was purchased from M/s. Vijaya Scientific Madurai. The paraffin wax melting point determined by supplier was between 56 and 60 °C. The reinforcement particles MWCNT and NBN were purchased from M/s. Sigma-Aldrich, Germany. The diameter and length of the MWCNT are 20 nm and 30 µm. The particle size of the NBN was 30 nm and the corresponding TEM images are shown in Fig. 1a and c.

Figure 1b and d shows the ring diffraction (interference (fringe) pattern) of MWCNT and NBN. This ring pattern is clearly showing Miller Indices named according to the interplanar spacing of MWCNT and NBN. However, these images also revealed presence of many rings corresponding to a set of atomic planes. The diffraction fingers are in regular, displaying that the level of impurities of the particles are less.

Fabrication of hybrid PCMs

Paraffin wax (5 g) is taken by mass % basis and heated in a water bath for 70 °C and then kept in a magnetic stirrer. The desired mass % of MWCNT and NBN was added in the liquid state of paraffin wax. Continuous stirring was performed for 1 h to reduce the agglomeration of reinforcing particles in the molten paraffin wax. To get a better/homogeneous dispersion of the reinforcing particles, the paraffin wax with additives was sonicated using ultrasonicator for another 2 h, similarly all four (99.5 paraffin wax + 0.5 MWCNT, 99.5 paraffin wax + 0.5 BN, 99 paraffin wax + 0.5 MWCT + 0.5 BN and 98 paraffin wax + 1 MWCNT + 1 BN) samples were prepared by same procedure and the schematic representation is shown in Fig. 2. For further analysis, the hybrid PCMs are cylindrical formed and as exhibited in Fig. 3.

Characterization of hybrid PCMs

The surface morphology and distribution of the reinforcing particles were analyzed using FE-SEM. The phase change of the MWCNT and NBN was evaluated using X-Ray diffraction technique. By using DSC analysis, the thermal properties such as melting point, solidification point and latent heat

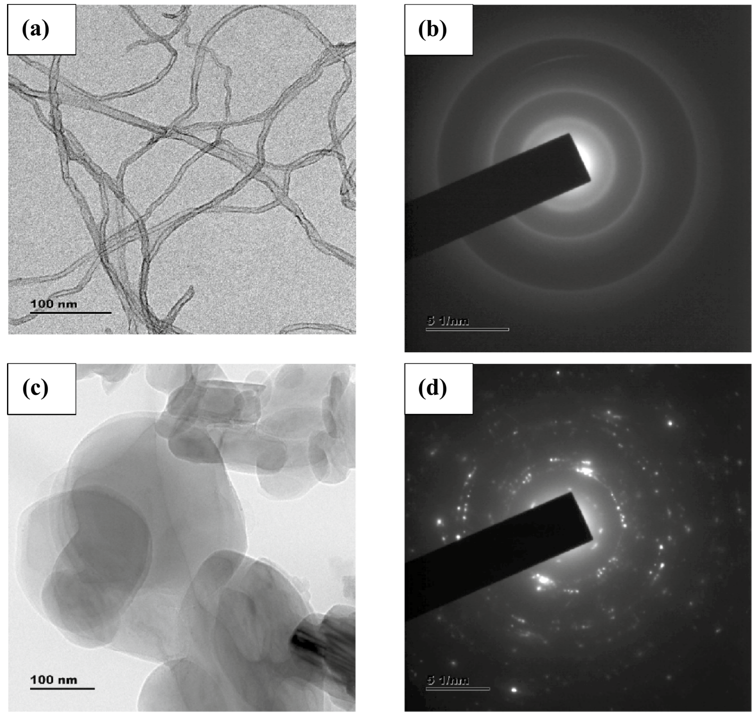

Fig. 1 TEM images of **a** MWCNT and **c** NBN and **b** and **d** the corresponding diffraction pattern

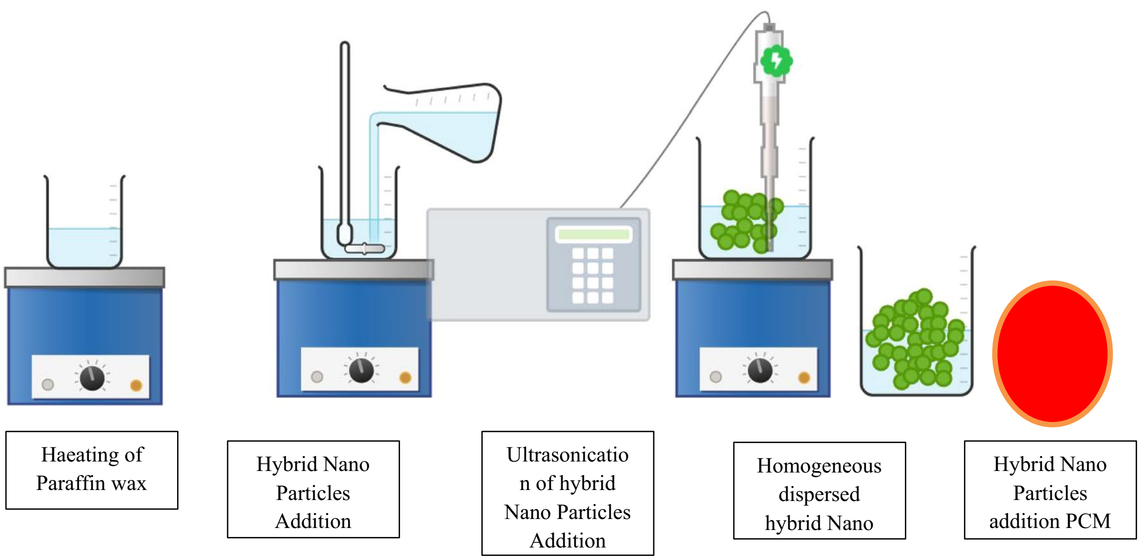

Fig. 2 Schematic representation process flow of fabrication of hybrid PCMs

are evaluated. The degradation of material loss percentage was assessed using TGA. Further the thermal stability of the hybrid PCMs was determined by same TGA, with higher operating temperatures. Afterwards, the thermal conductivity of the hybrid PCMs was evaluated using KD2 Pro thermal properties analyzer.

Results and discussion

Surface morphology

The surface morphology obtained through FE-SEM of fabricated hybrid PCMs is displayed in Fig. 4a, b, c and d. Paraffin wax melting point is low; hence images were captured with low resolution. Before performing the

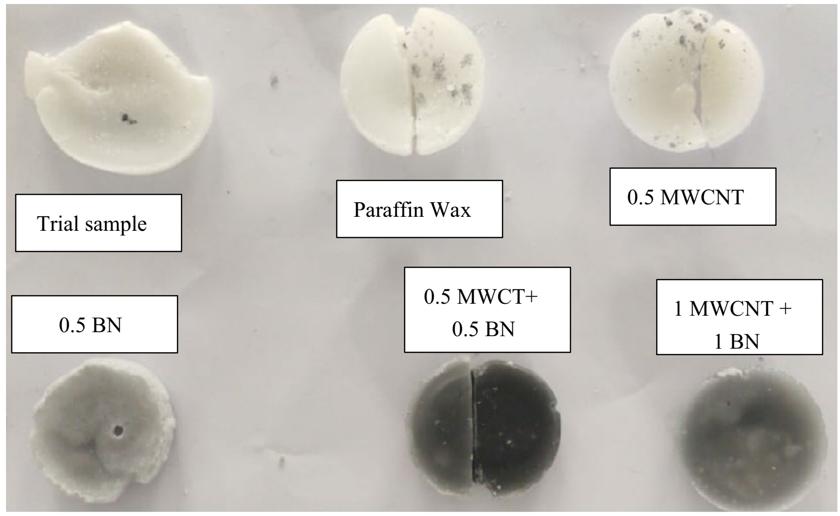

Fig. 3 Photographic view of different fabricated PCMs

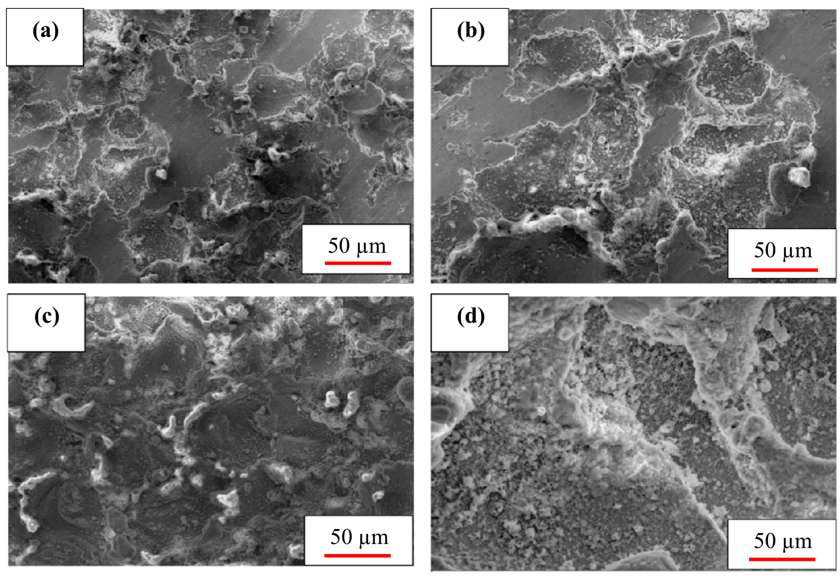

Fig. 4 FE-SEM Surface morphology of different PCMs **a** 99.5 paraffin wax + 0.5 MWCNT, **b** 99.5 paraffin wax + 0.5 NBN, **c** 99 paraffin wax + 0.5 MWCT + 0.5 NBN and **d** 98 paraffin wax + 1 MWCNT + 1 NBN

experimentation, the PCMs were coated with gold particles for better conductivity. FE—SEM images clearly illustrate that the shape of the paraffin wax was a pack of branches [10–14]. However, with increasing mass % of MWCNT and NBN, the bundles got worn out. On the other hand, increasing the mass % of MWCNT and NBN with proper ultrasonication, prevented agglomeration of the particles. It influenced the interaction between the paraffin wax and reinforcing particles and hence enhancement of physical and thermal properties.

XRD analysis

Figure 5a shows the XRD spectrum of MWCNT and NBN with strong crystalline peak observed at 23.32–43.52° for MWCNT and 24.22–41.25° for NBN [15]. Other two moderate diffraction patterns were observed at 45.35–55.12° for MWCNT and 32.03–45.35° for NBN. The intensity of the peaks clearly indicated the presence of MWCNT and NBN and it was also confirmed by the JCPDS card no. 26–1079 and 34–0421. XRD pattern of hybrid PCM such as Paraffin Wax + 0.5 MWCNT + 0.5 BN and Paraffin Wax + 1.0 MWCNT + 1.0 BN is shown in Fig. 5b. It is illustrated that MWCNT and BN was homogeneously dispersed in paraffin wax. The intensity of peaks reveals the different proportions of MWCNT and NBN in paraffin wax.

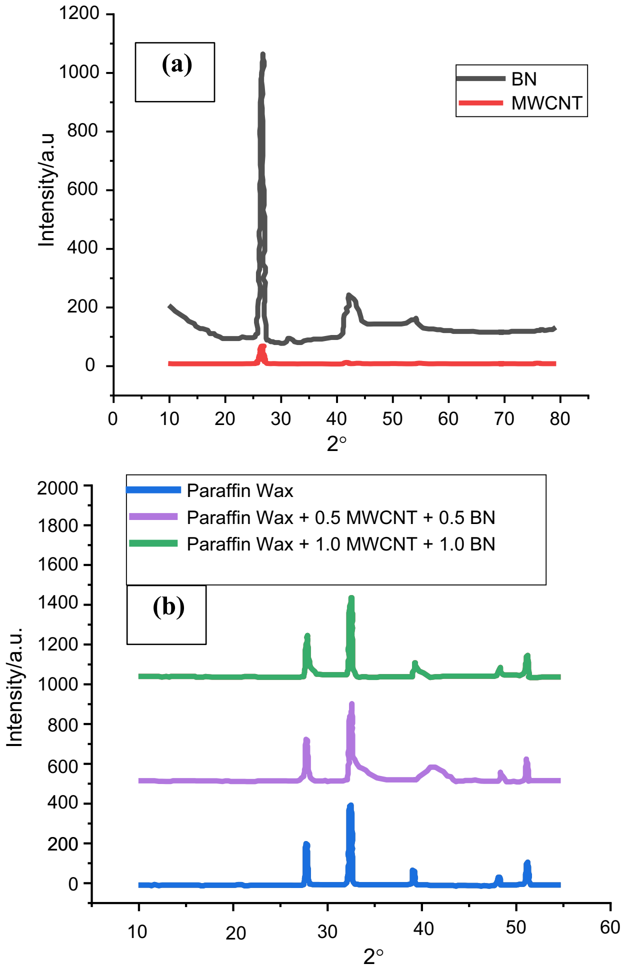

Fig. 5 XRD spectrum **a** MWCNT and BN **b** Paraffin Wax + MWCNT + BN

DSC analysis

Figure 6a, b, c, d and e shows the DSC curves analysis of hybrid PCMs containing MWCNT and NBN concentrations. It illustrates that MWCNT with paraffin wax and NBN with paraffin wax slightly reduced the melting onset temperature and hence the solidification onset temperature. However, the hybrid nano-particles MWCNT and NBN enhanced the peak melting temperature and minimally decreased the peak solidification temperature of paraffin wax. Increasing the mass % of MWCNT and NBN provided better variations of the composition of the paraffin and resulted in significant change in phase change temperature. Two different kinds of peaks were observed in DSC analysis and it revealed that the initial peak contained heating, because of solid-to-solid transformation of hybrid PCMs, whereas one large peak was observed, that illustrated solid to liquid phase transformation of hybrid PCMs [16–19]. However, during solid-to-solid phase transformation, the crystalline structure changed to amorphous structure and it clearly correlated with XRD spectrum. On the other hand, the lower peak indicated that the hybrid PCMs with MWCNT and NBN provide better heat changes. This phenomenon is mainly happening because of excellent thermal conductivity of MWCNT and NBN and leading to better interface between the nano-particles and paraffin wax. The homogeneous dispersion of nano-additives in hybrid PCMs will lead to changes in the heat capacity of the PCM which is relatively uniform.

Performance of hybrid PCM is mainly attributed to the surface properties of MWCNT, NBN and paraffin wax. Increasing the mass % of MWCNT and NBN, improved the thermal conductivity of paraffin wax, leading to rapid heat transfer and hence early onset of phase change due to lower phase change temperature. Addition of nano-particles with high thermal conductivity changes the performance of phase change onset temperature of hybrid PCMs. Solid to liquid phase transformation is significant for paraffin wax since large amount of heat energy in the form of latent heat can be stored using this. Increasing the mass % of MWCNT and NBN will reduce the melting point of paraffin wax from 64.70 to 62.52 °C. The solidification temperature of paraffin wax increased while increasing the mass % of MWCNT and NBN and became 56.01 to 60.13 °C respectively. The thermal properties of MWCNT-BN/paraffin PCM samples are displayed in Table 1. From the above results, it is clearly observed that increasing the mass % of MWCNT and NBN decreased the melting onset and increased the solidification onset temperature, which is due to the nano-additive particles acted as nucleation agents and reduced the supercooling of paraffin wax. The outcome of the above results, articulate the addition of mass % of reinforcing particles will directly influence the thermal storage capacity of PCMs [19–22].

TGA analysis

Figure 7 shows the TGA analysis graph of hybrid PCMs and it illustrates that the same trend was obtained for all the samples. For this experiment, 5.5 mg of paraffin wax with MWCNT and NBN were exposed to average heating rate of 10 °C min^{-1} from 30 to 450 °C in nitrogen environment. Based upon the previous researchers' recommendations, the heating rate (10 °C min^{-1}) was selected. For hybrid PCMs with MWCNT and NBN, the degradation temperature increased slightly at the beginning and end of the curve. In the case of pure paraffin wax, the thermal degradation started at 140 °C, 153 °C, 157 °C, and 163 °C and ended at 348 °C, 364 °C, 386 °C, and 397 °C, respectively. The similar trends were observed for other samples such as (a) 99.5 paraffin wax + 0.5 MWCNT, (b) 99.5 paraffin wax + 0.5 NBN, (c) 99 paraffin wax + 0.5 MWCT + 0.5 NBN and (d) 98 paraffin wax + 1 MWCNT + 1 NBN, respectively. The paraffin wax was

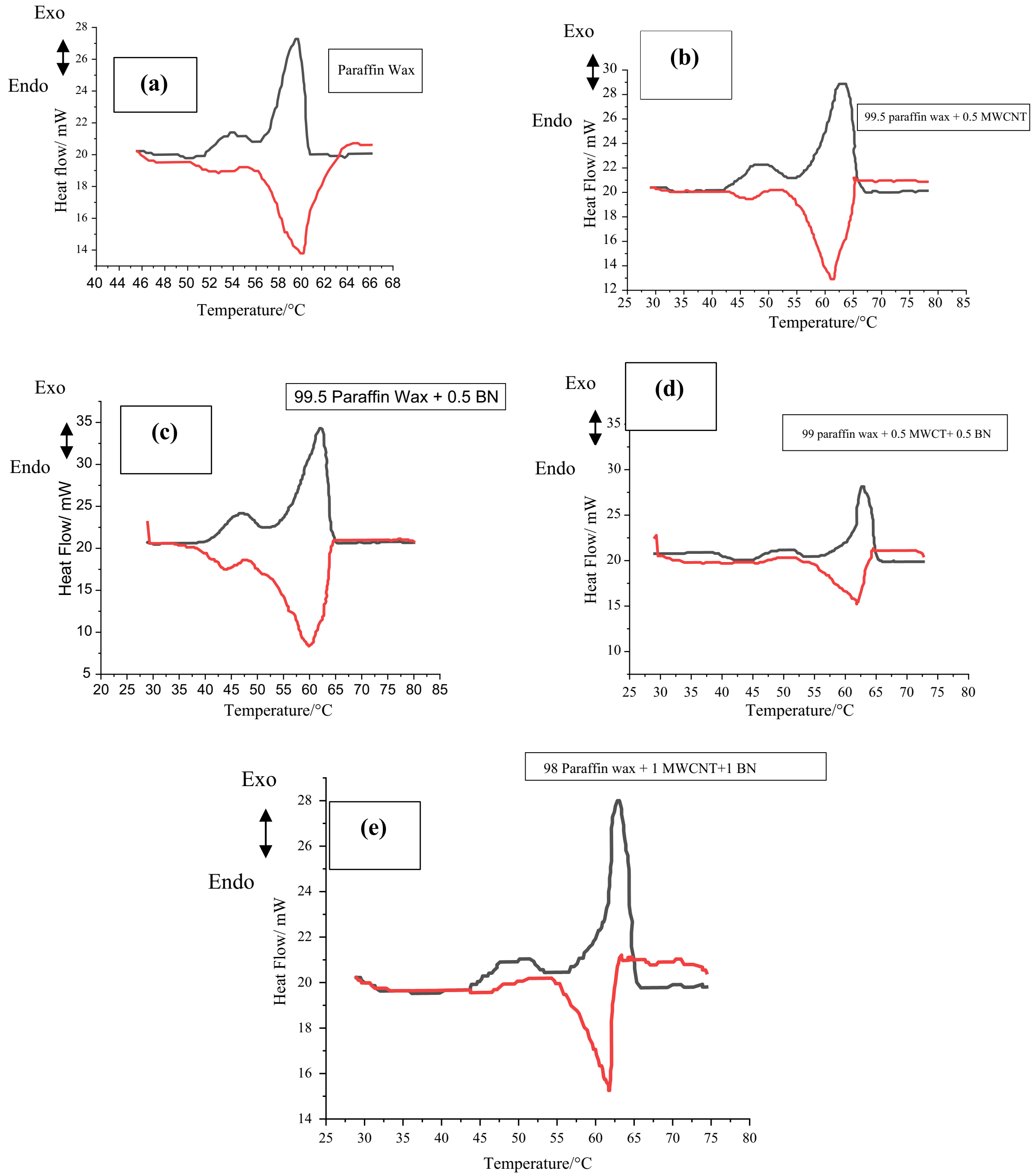

Fig. 6 DSC Analysis for hybrid PCMs **a** Paraffin wax, **b** 99.5 paraffin wax + 0.5 MWCNT, **c** 99.5 paraffin wax + 0.5 BN, **d** 99 paraffin wax + 0.5 MWCT + 0.5 BN and **e** 98 paraffin wax + 1 MWCNT + 1 BN

removed at 260 °C, and it completely degraded at 450 °C for all the other hybrid PCMs. It is revealed that the addition of MWCNTs and NBN has impact on the decomposition of paraffin wax, and the results agree with previous research findings [23–26].

Evaluation of thermal stability

Thermal cyclic testing was carried out to determine the thermal stability of PCMs. In this experiment, the samples were heated above its melting point using hot water bath and cooled at room temperature naturally by cold water bath. 100 cycles were carried out in this investigation. Hybrid PCMs possessed good stability and the thermal conductivity was determined for uncycled and

Table 1 Thermal properties of MWCNT-BN/paraffin PCM samples

Samples	Melting point/°C	Melting onset temperature/°C	Solidification point /°C	Solidification onset temperature/°C	Latent heat during melting/ kJ kg^{-1}	Latent heat during Solidification/ kJ kg^{-1}
Paraffin wax	64.70	57.20	56.01	61.50	139.50	132.30
99.5 paraffin wax + 0.5 MWCNT	64.00	56.25	57.10	65.00	138.60	130.10
99.5 paraffin wax + 0.5 BN	63.80	56.00	58.25	65.20	132.35	125.60
99 paraffin wax + 0.5 MWCT + 0.5 BN	63.20	55.25	59.15	65.50	125.60	110.25
98 paraffin wax + 1 MWCNT + 1 BN	62.52	55.05	60.13	66.10	110.20	96.50

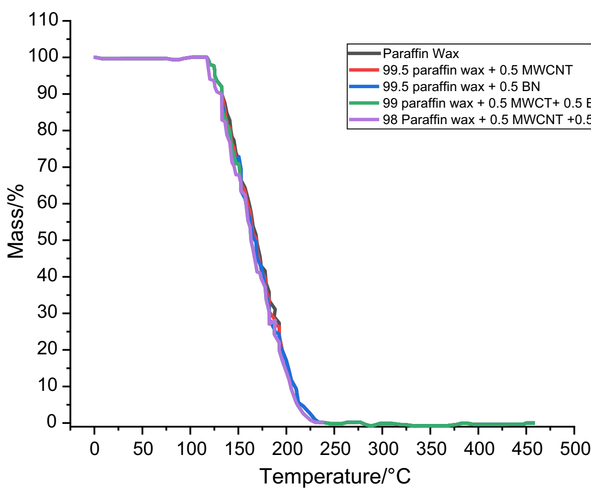

Fig. 7 TGA Analysis for hybrid PCMs

Table 2 Thermal Stability values for both uncycled and cycled condition

Sample	Thermal conductivity/ $Wm^{-1} K^{-1}$	
	Uncycled condition	Cycled condition
Paraffin wax	0.250	0.250
99.5 paraffin wax + 0.5 MWCNT	0.355	0.340
99.5 paraffin wax + 0.5 BN	0.395	0.385
99 paraffin wax + 0.5 MWCT + 0.5 BN	0.416	0.398
98 paraffin wax + 1 MWCNT + 1 BN	0.456	0.420

cycled conditions of (a) Paraffin wax, (b) 99.5 paraffin wax + 0.5 MWCNT, (c) 99.5 paraffin wax + 0.5 NBN, (d) 99 paraffin wax + 0.5 MWCT + 0.5 NBN and (e) 98 paraffin wax + 1 MWCNT + 1 NBN after 100 cycles and displayed in Table 2. It illustrated that increasing the mass % of MWCNT and BN leads to increase in the thermal conductivity for both uncycled and cycled condition. In comparison with uncycled PCMs, cycled PCMs have lower thermal conductivity due to nano-composite rearrangement in paraffin wax.

Evaluation of thermal conductivity

The thermal conductivity of the hybrid PCMs is displayed in Fig. 8a, b and c and it illustrates that increasing the mass % of MWCNT and NBN will significantly increase the thermal conductivity (Refer Fig. 8c). The thermal conductivity is mainly depending on how the nano-particles are dispersed in PCM [27]. The dispersion mainly depended on the duration of stirring and ultrasonication. The effective ultrasonication provided soft interface or suspension between the hybrid nano-particles in PCM and it created network to transfer heat in all the directions of PCM quickly. According to Fig. 8a, the thermal conductivity of pure paraffin wax was 0.18 $Wm^{-1} K^{-1}$ and increased from 0.20 $Wm^{-1} K^{-1}$ to 0.31 $Wm^{-1} K^{-1}$ for 98 paraffin wax + 1 MWCT + 1 NBN hybrid PCM. The thermal conductivity increased linearly while increasing the mass % of nano-additive particles [27]. This happened because of the homogeneous dispersion of nano-particles, in the PCMs. The thermal distribution of hybrid PCMs is displayed in Fig. 8b. Three colors are identified such as blue, green and red. Blue pertained to the maximum level of thermal conductivity, green moderate level and red minimum level of thermal conductivity due to the influence of nano-particles in paraffin wax [28]. According to Fig. 8c ternary diagram, 1 MWCNT + 1 NBN reflects maximum thermal conductivity distribution of PCM.

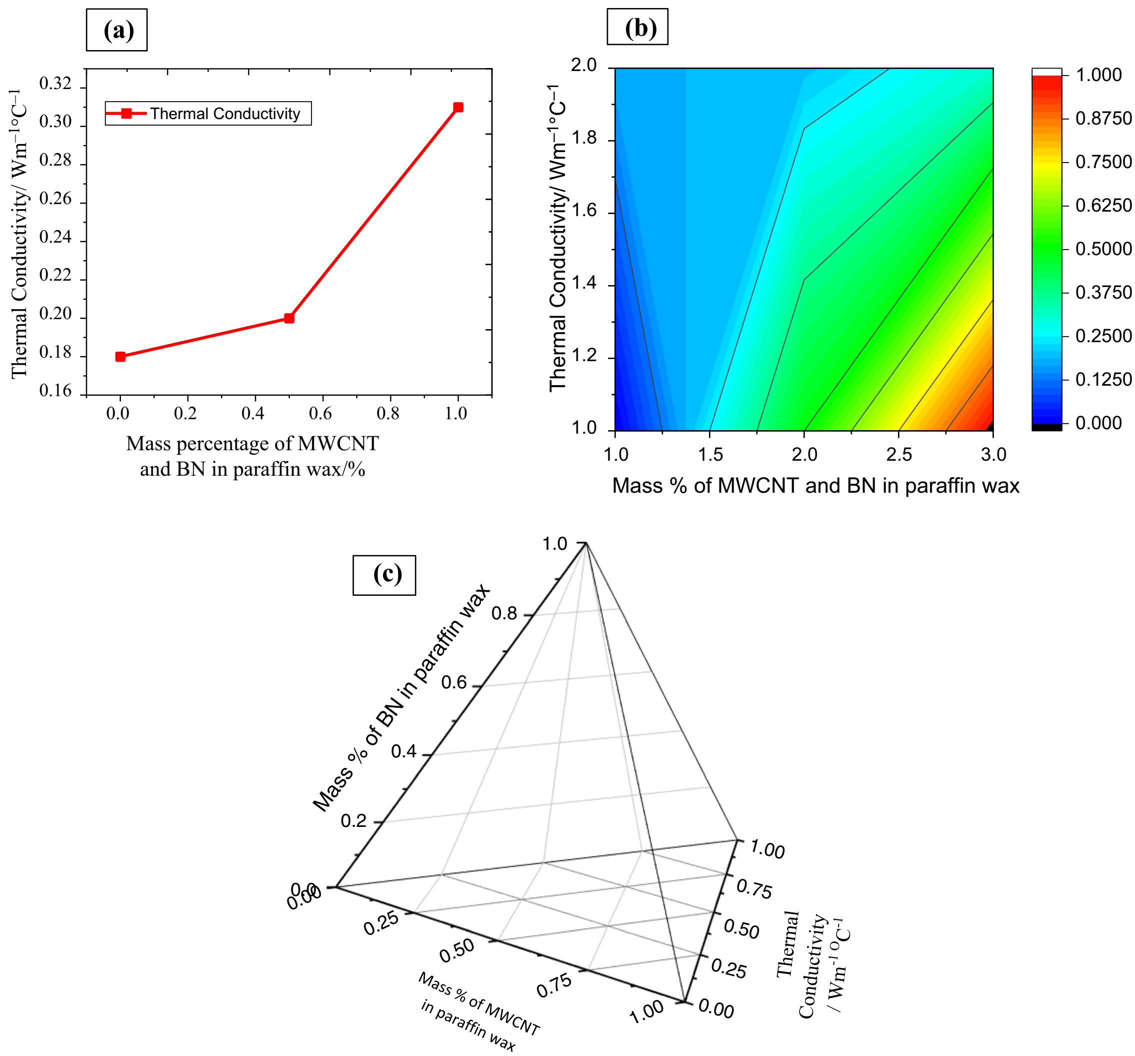

Fig. 8 Thermal Conductivity of hybrid PCMs **a** Line graph, **b** Thermal Distribution for hybrid PCMs and **c** ternary diagram of nano-particle distribution for hybrid PCMs

Conclusions

- The size of the nano-particles was ensured using transmission electron microscope (TEM). The diffraction fingers are regular indicating that the level of impurities in the particles is less.
- FE—SEM images clearly illustrated that the shape of the paraffin wax is a pack of branches. However, with increasing mass % of MWCNT and NBN, the bundle like structure got worn out.
- Increasing the mass % of MWCNT and NBN reduced the melting point of paraffin wax from 64.70 to 62.52 °C. The solidification temperature of paraffin wax increases while increasing the mass % of MWCNT and NBN and reached 56.01–60.13 °C, respectively.
- Thermal degradation started at 140 °C, 153 °C, 157 °C, and 163 °C and ended at 348 °C, 364 °C, 386 °C, and 397 °C, respectively, for the samples.
- The thermal conductivity of pure paraffin wax was $0.18 Wm^{-1} K^{-1}$ and it increased from $0.2 Wm^{-1} K^{-1}$ to $0.31 Wm^{-1} K^{-1}$ for hybrid PCMs.

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