PREPARATION OF NANO – SIO_2/PARAFFIN PCM SAMPLES AND MEASURING ITS PARAMETERS

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Abstract: The impact of low mass% SiO2 nano particles on the thermal characteristics of paraffin wax is examined in this paper in the context of solar thermal energy storage applications. 0.0 mass%, 0.5 mass%, 1.0 mass%, and 2.0 mass% of SiO2 nano particles in paraffin wax were used to make the four nano-SiO2/paraffin PCM samples. To evaluate their thermal properties and physical structure, the produced nano-SiO2/paraffin PCMs were investigated using a variety of experimental approaches. The analysis showed that the fusion of SiO2 nano particles with paraffin wax was homogeneous, and they increased the paraffin wax's thermal conductivity to 12.78%, 22.78%, and 33.34%, respectively, with the aforementioned mass percentage of SiO2 nano particles Additionally, the SiO2 nano particle dispersion improved the melting and solidification temperatures of the paraffin wax while extending its thermal degradation without altering its chemical composition. However, as the mass percentage of the nanoparticles increased, the latent heat of the paraffin wax fell; this decrease is notable for paraffin wax that contains more than 1.0 mass percentage of SiO2 nanoparticles. Overall, the results showed that paraffin wax combined with the lowest mass% of SiO2 nanoparticles significantly improved its thermal characteristics and was suitable for increased thermal energy storage.

Keywords: Thermal energy storage; phase change material; characterization of phase change material; nano-SiO2/paraffin; SiO2 nano particles.

INTRODUCTION

When designing solar thermal systems like solar water heaters, solar air warmers, solar distillation, solar driers, and other sun thermal applications, it is extremely difficult to provide a constant supply of thermal energy during times of demand (Da Cunha and Eames 2016; Kaygusuz 1999). Due to occasionally unpredictable and changing weather and climatic circumstances, solar energy is only available for a brief period of time (Kaygusuz 2003; Sharshir et al. 2017). In order to supply thermal energy regardless of the solar energy's dynamic behaviour, it is therefore required to construct solar thermal systems with an appropriate layout., due to their higher energy density, which ultimately contributes to the storage of a significant amount of thermal energy in the form of latent heat (Farid et al. 2004; JianShe et al. 2019; Karaipekli, Sari, and Kaygusuz 2009; Sari et al. 2013). In order to assess the potential of diverse types of phase change materials, including organic and inorganic phase change materials, to be used as energy storage materials, substantial study was done on these materials (Cabeza et al. 2011; Ibrahim et al. 2017). Paraffin wax, in contrast, has been tested as an energy storage material by the majority of researchers due to its superior large energy density, low vapour pressure, good chemical and thermal stability, reduced need for supercooling, non-toxicity, widespread availability, and

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low cost (Shukla, Kant, and Sharma 2017; Xiao, Zhang, and Sharma 2018). Despite the better qualities of paraffin noted above, they fall short due to their poor thermal conductivity (Anghel et al. 2014; Karaipekli et al. 2017). The literature had proposed methods for increasing the thermal conductivity of paraffin wax, including adding porous materials to paraffin (Zhong et al. 2010a, 2010b; Huang et al. 2017), incorporating high conductivity (metallic and non-metallic) particles, and submerging highly conductive metallic fins in PCMs (Khodadadi and Hosseinizadeh 2007). The majority of researchers are interested in the dispersion of highly conductive nanoparticles in paraffin wax among the known techniques since it significantly improves the thermal conductivity of base PCM (Ahmed et al. Liu et al. 2015; Ibrahim et al. 2017). According to Wang et al. (2009), the thermal conductivity ratios increased to 35–40% with the dispersion of 2.0% mass fraction of multi-walled carbon nano tubes in paraffin wax. The influence of 0.3, 0.5, and 1.0 mass percentages of carbon nano tubes on the thermal conductivity of the expanded perlite/paraffin composite phase transition material was experimentally investigated by Karaipekli et al. (2017They acknowledged that the composite's thermal conductivity was increased to 113.3% using

Carbon nano tubes make up 1.0 mass percent. The thermal characteristics of paraffin were improved by the addition of zinc oxide nano particles, and it was argued that this modification made paraffin ideal for solar thermal energy storage applications (Ahan and Paksoy, 2017). It is clearly understood that the majority of the literature has focused on the dispersion of carbon-based particles in base PCMs (JianShe et al. 2019; Karaipekli et al. 2017; Wang et al. 2009; Yu et al. 2008) as well as nano particles of metallic and metal oxides (Kalaiselvam, Parameshwaran, and Harikrishnan 2012; Lin and Al-Kayiem 2012 However, the effect of non-metallic nano particles on the thermal characteristics of paraffin wax is rarely discussed in the literature that is currently available. When Yang et al. (2014) investigated how silicon nitride nano particles affected the thermal characteristics of paraffin, they clearly demonstrated that the thermal conductivity of paraffin improved with an increase in silicon nitride nano particles. in terms of silicon nitride nano particle mass %. Since that time, a thorough examination of paraffin's thermal characteristics in conjunction with any other silicon-based nano particles is hardly ever found in contemporary literature. This encourages in-depth research into the potential of low mass% SiO_2 nanoparticles utilized as a nano additive in paraffin wax to improve its thermal characteristics. The goal of this work is to experimentally analyze the various thermo physical properties of paraffin wax by adding nano-SiO_2 particles in three different mass fractions. In doing so, this paper becomes novel in its exploration of the possibility for finding the potential use of nano-SiO_2/paraffin as nano-enhanced phase change material in solar heating applications. The experimental outcomes of nano-SiO_2/paraffin PCMs are then contrasted with those of paraffin wax in its purest form.

2. METHODOLOGY AND EXECUTION

It is more affordable than any other kind of technical grade paraffin with the appropriate thermo physical qualities, the commercial-grade paraffin wax used in candle production is chosen as the basis PCM in this work. From Jayam Chemicals in Coimbatore, paraffin wax was purchased. Paraffin wax can be employed as a thermal energy storage medium in solar heating applications since it has a melting temperature that falls between 56°C and 60°C, according to the source. Sisco Research Laboratories Pvt. provided SiO_2 nano particles with an average size of 15 nm. Mumbai, Ltd.

Table 1: Displays the thermo physical characteristics of SiO_2 nano particles and pure paraffin wax.

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Properties	Paraffin wax	SiO_2 nanoparticles
Melting point (°C)	63.74	1713
`	57.01	-
Latent Heat (kJ/kg)	140.2	-
Thermal Conductivity(W/mK)	0.180	1.5

Preparation of nano-sio$_2$/paraffin PCM

By dispersing various mass% of nano-SiO2 particles in pure paraffin wax, the nano-SiO2/paraffin PCM was created. To avoid particle agglomeration and an unexpected rise in material costs, the researchers advise dispersing low mass nano particles in the range of 0.2–4%. (Cai et al. 2011; Kumar and Mylsamy 2018; Wang et al. 2009). Therefore, the mass% of nano-SiO2 particles in paraffin wax used in this experiment is 0%, 0.5%, 1.0%, and 2.0%. Two steps were used to prepare the nano-SiO2/paraffin PCM samples (Amin et al. 2017). The paraffin wax was heated to 70°C in a hot water bath at first, and it was then held on a magnetic stirrer. The needed mass percent of nano-SiO2 particles was then gradually added to the molten paraffin wax, and the nano composite PCM was heated and agitated simultaneously for an hour. To prevent nano particle agglomeration and create a consistent, homogenous mixture, the mixture was further placed in an ultrasonic vibrator and solicited for an additional two hours. In Table 2, the compositions of samples made with various nano-SiO2/paraffin ratios are displayed. For further characterisation, the materials were shaped into cylindrical forms as illustrated in Figure 1.

Characterization of nano-SiO_2/paraffin PCM

Joule Wax Industry Co., Ltd. (Shanghai, China) supplied the paraffin for the PCM experiment, and its phase change temperature was 28 °C. Shanghai Macklin Biochemical Technology Co., Ltd. provided the amorphous mesoporous silica (Shanghai, China). Mesoporous silica, represented by MS1, MS2, and MS3, had average pore diameters of 15 nm, 30 nm, and 50 nm, respectively. For MS1, MS2, and MS3, the average specific surface areas were 230 m2/g, 180 m2/g, and 150 m2/g, respectively, with an up-and-down variation of 30 m2/g. MS had a pH value between 5-7, a loss on ignition at 1000 °C of no more than 2%, and a purity of greater than 99.5%.

2.2. Preparation

The SS-PCMs were prepared via direct adsorption. A predetermined amount of paraffin was weighed and put into a beaker, which was heated in a drying box at a constant temperature of 50 °C. The paraffin was applied to mesoporous silica with various pore diameters after it had completely melted. The beaker containing PCM composites was placed in a hot water bath at a temperature of 50 °C and agitated for 30 min at 300 r/m to ensure that the paraffin was spread equally in the mesoporous silica. The PCM composites were then chilled at 4 °C in a refrigerator. The mass ratios of paraffin and mesoporous silica in SS-PCM samples with various mass percentages of paraffin were generated.

The generated nano-SiO2/paraffin PCM samples (0%, 0.5%, 1.0%, and 2.0% by mass of nano-SiO2) were further evaluated with various devices to examine their morphology and assess their thermal characteristics. Using a Zeiss Sigma FESEM, which uses an Everhart-Thornley type (SE2) detector for imaging, the surface morphology and microstructure of the pure paraffin and nano-SiO2/paraffin PCMs were examined. With the use of Melvern's Zetasizer AT, the size distribution of the

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commercially available SiO2 nanoparticles was confirmed in terms of their diameters. PerkinElmer's Spectrum 2 Fourier Transform Infrared Spectrometer was used to examine the generated samples' chemical bonding and molecular behaviour. Thermal characteristics,such as latent temperatures of solidification, melting points, and PCMs) (DSC). The DSC 6000 with Pyris 6 DSC, made by PerkinElmer Inc., was used in conjunction with DSC. PerkinElmer Inc.'s TGA 4000 Thermo gravimetric Analyzer was used to evaluate the weight loss % and degradation temperature of nano-SiO2/paraffin PCMs in order to ascertain their thermal stability at higher working temperatures. The KD2 Pro thermal properties analyzer, which is primarily operated under the transient line heat source method, was used to measure the thermal conductivity of the samples.

Table2. Composition of nano-SiO_2/paraffin PCM samples.

S.No.	Samples	Composition in mass (grams)
1	0 mass% of nano-SiO_2/paraffin PCM	157 paraffin wax
2	0.5 mass% of nano-SiO_2/paraffin PCM	150.25 paraffin wax +0.75 SiO_2 nanoparticles
3	1.2 mass% of nano-SiO_2/paraffin PCM	150.5 paraffin wax +1.5 SiO_2 nanoparticles
4	2.2 mass% of nano-SiO_2/paraffin PCM	149 paraffin wax

Figure 1. Photograph of the prepared nano-SiO2/paraffin PCM samples.

Results and Discussion

FESEM analysis

Field emission scanning electron microscopy (FE-SEM) is an advanced technology used to capture the microstructure image of the materials. FE-SEM is typically performed in a high vacuum because gas molecules tend to disturb the electron beam and the emitted secondary and backscattered electrons used for imaging.

Shown in Figure2. Low-resolution scanning is used instead of scanning electrons in order to prevent melting of paraffin wax. The samples were first given a gold coating to make them more conductible. The materials were then photographed using a FESEM by being bombarded with electrons. The paraffin wax image shows that its structure is made up of a bundle of strata. With the increase in the

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mass percentage of nano-SiO2 particles in the base PCM, these bundles disappeared. Additionally, the photos demonstrated that improper physical interaction between the nanoparticles and the base PCM occurred during the careless aggregation of the nanoparticles.

How does a FESEM function:

Electrons are liberated from a field emission source and accelerated in a high electrical field gradient. Within the high vacuum column these so-called primary electrons are focused and deflected by electronic lenses to produce a narrow scan beam that bombards the object. As a result secondary electrons are emitted from ech spot on the object. The angle and velocity of these secondary electrons relates to the surface structure of the object. A detector catches the secondary electrons and produces an electronic signal. This signal is amplified and transformed to a video scan-image that can be seen on a monitor or to a digital image that can be saved and processed furth

Particle size analysis

Particle size analysis, particle size measurement, or simply particle sizing, is the collective name of the technical procedures, or laboratory techniques which determines the size range, and/or the average, or mean size of the particles in a powder or liquid sample.

Particle size analysis is the part of particle science, and it is generally carried out in particle technology laboratories. The particle size measurement is typically achieved by means of devices, called Particle Size Analyzers (PSA), which are based on different technologies, such as high definition image processing, analysis of Brownian motion, gravitational settling of the particle and light scattering (Rayleigh and Mie scattering) of the particles. The particle size can have considerable importance in a number of industries including the chemical, food, mining, forestry, agriculture, cosmetics, pharmaceutical, energy, and aggregate industries.

nano-SiO2 particles were also examined to confirm that they had the size distribution that the supplier had specified. The bar chart of the size distribution created using Zetasizer is shown in Figure 3. It was observed that the distribution of particles with a diameter of 15–19.9 nm exhibited the highest percentage at 21.4%, followed by those with a diameter of 20–24.9 nm at 18.2%. Consequently, it meets the requirements of the suppliers (15 nm average size distribution of SiO2 nanoparticles). Additionally, the accuracy of using the aforementioned nanoparticles size for numerous characterization tests was confirmed by this particle size analysis.

FT-IR analysis

The surface of the pure paraffin wax and the various nano-SiO2/paraffin PCM samples was scanned with the aid of a FESEM, and the pictures are shown in Figure 4. The outcomes of the FT-IR analysis performed on the paraffin wax and the various nano-SiO2/paraffin PCM samples are shown in Figure 4. With the inclusion of the nano-SiO2 particles in various mass percentages, the analysis showed that the peak wavelength of the paraffin wax did not vary significantly. Strong asymmetrical and medium symmetrical C-H stretching vibrations are shown by two strong peaks at wavelengths 2848 cm-1 and 2916 cm-1, respectively. Similar to that,the abrupt peaks at 729 cm and1462cm indicates medium Rocking medium scissor vibration of C-H and skeleton vibration of C-C respectively. The medium –amplitude, asymmetric stretching vibration of C-H is visible in the faint peak at 2956 cm1. All of the sample, including the sample, including sample of paraffin wax ,have the a aforementioned peaks (figure 4a-d) moreover, a weak peak of 1102 cm-1 has been found in the sample containing nano-sio2 particles of difference of mass%. this indicates the very strong a symmetrical SI-O-C stretching vibration and proves the strong bonding of Si02 nano particles is strong and concurrently did not disturb the chemical structure of paraffin fax.

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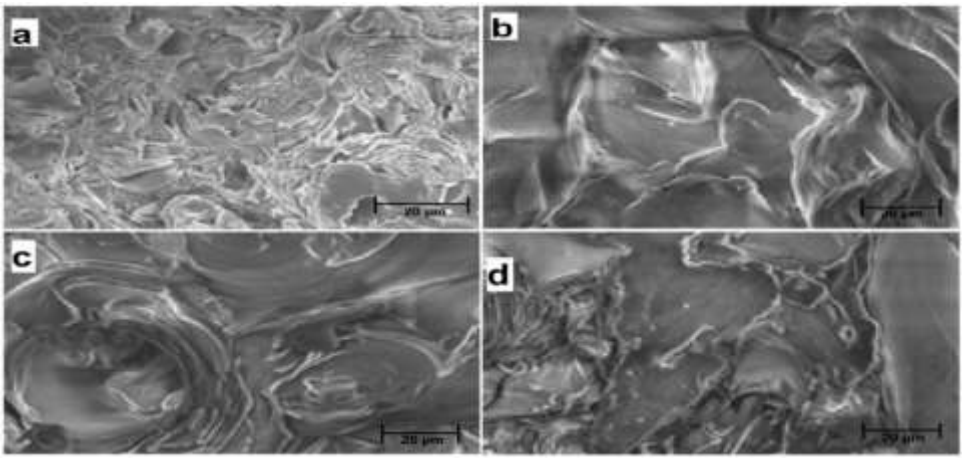

Figure 2. FESEM scanned images of the surface of the samples (a) paraffin wax (b) 0.5 mass% of nano-SiO2/paraffin PCM (c) 1.0 mass% of nano-SiO2/paraffin PCM (d) 2.0 mass% of nano-SiO2/paraffin PCM.

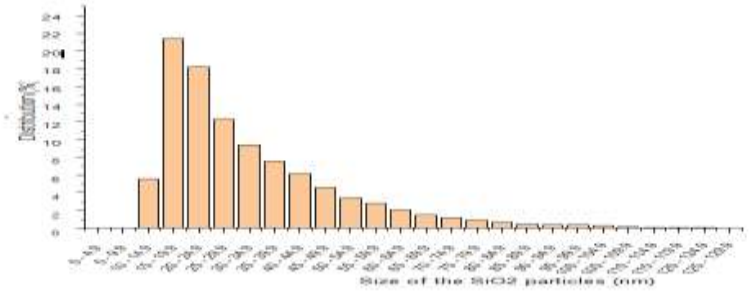

Figure 3. Size distribution of SiO2 nanoparticles used in the experiments.

DSC analysis

Figure 5 displays the differential scanning calorimetric data for the nano-SiO2/paraffin PCM samples. Two different types of peaks can be seen on the DSC plots of the samples. The PCMs' initial peak upon heating results from their transition from the solid to solid phase, whereas their greater peak shows their transition from the solid to liquid phase. The PCM's crystalline structure splits and transforms from one solid to another during this phase transition. The similar kind of plots were followed by Mohamed et al. (2017) and Murugan et al. (2018) when they explored paraffin wax with multi-walled carbon nano tubes and nano alumina, respectively. However, the primary element in the transition from the solid to liquid phase is SiO2 nano particles served as the paraffin wax's nucleation agents and prevented it from super cooling. However, the latent heat of pure paraffin wax decreased according to the mass percent ofSiO2.

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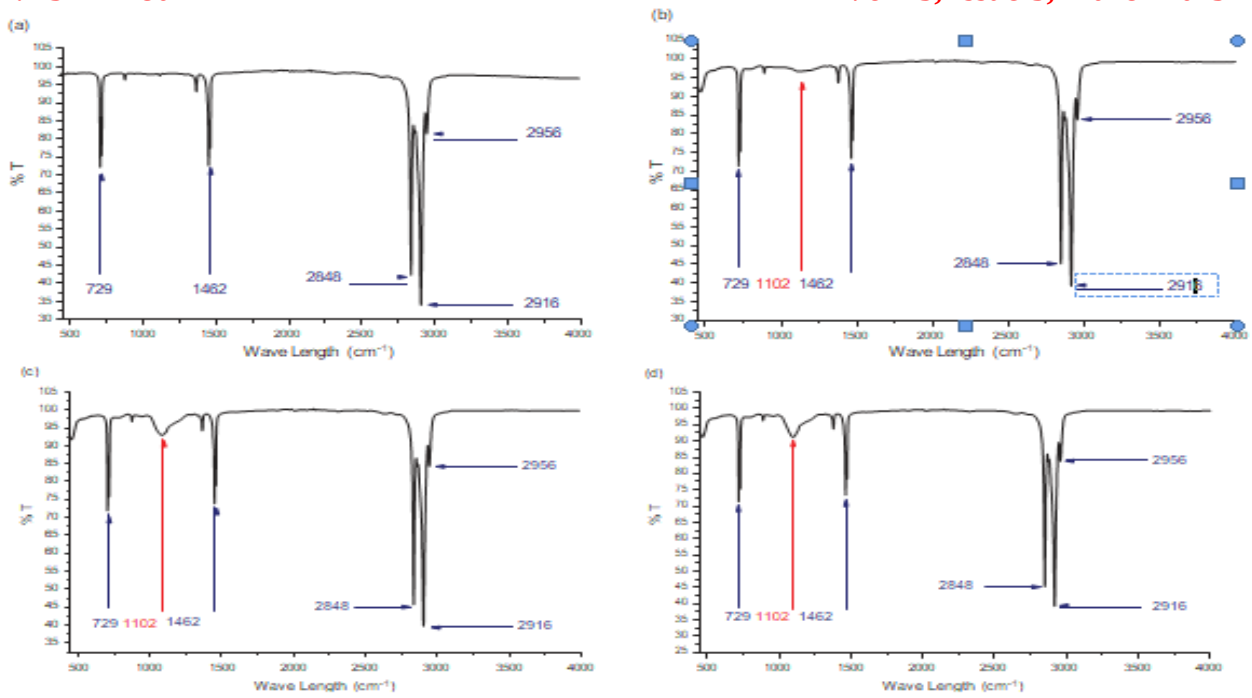

Figure 4.FT-IR analysis of the samples (a) paraffin wax (b) 0.5 mass% of nano-SiO2/paraffin PCM (c) 1.0 mass% of nano-SiO2/ paraffin PCM (d) 2.0 mass% of nano-SiO2/paraffin PCM.

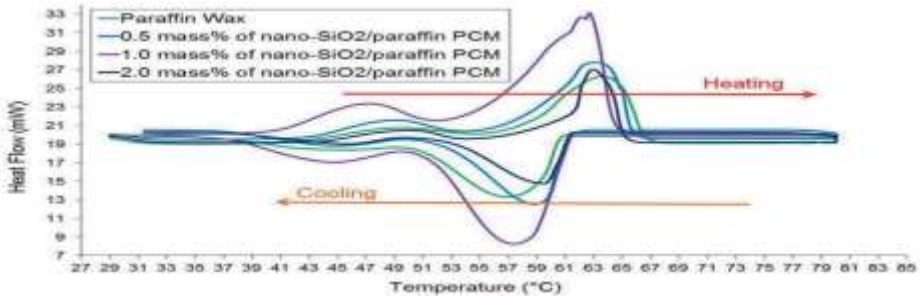

Table 3. Thermal properties of nano-SiO_2/paraffin PCM samples.

Samples of nano-SiO_2/paraffin PCMs	Melting point (°C)	Latent heat during melting (kJ/kg)	Solidification point (°C)	Latent heat during solidification (kJ/kg)
0 mass% of nano-SiO_2/paraffin PCM	65.74	142.2	58.01	136.6
0.5 mass% of nano-SiO_2/paraffin PCM	64.1	139.2	58.15	131.3
1.0 mass% of nano-SiO_2/paraffin PCM	63.72	135.0	58.54	129.2
2.0 mass% of nano-SiO_2/paraffin	63.08	107.3	59.41	100

Nanoparticles. Particularly, the reduction in latent heat of paraffin is prominent with 2.0 mass% of SiO_2 nano particles. Likewise, the reduction in latent heat did not follow the linearity and it may be due to the degree of mixing and agglomeration of SiO_2 nano particles in base PCM. The identical trend is reported in the work of Karaipekli et al. (2017). Hence, it must be kept in mind while the

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selection of mass% of the nano particles for a thermal storage application as considerable increase in mass% of nano particles deteriorates the storage capacity of the PCMs.

TGA analysis

Thermo gravimetric analysis of the paraffin wax and the nano-SiO_2/paraffin PCM samples were accompanied, by heating 5.5 mg of each sample at the rate of 10°C/min from 30°C to 450°C in nitrogen atmosphere. The heating rate of 10°C has been selected based on the similar previous researches (AlMaadeed et al. 2015; Mehrali et al. 2013). Figure 6 shows the result of the TGA analysis. It can be perceived that the plot shows the same trend for all the samples. However, the beginning and ending points of degradation temperature have been considerably increased by the addition of SiO_2 nanoparticles with paraffin wax. The thermal degradation initiated at 142°C, 157°C, 159°C, and 160°C and ended at 244°C, 262°C, 266°C, and 268°C for paraffin wax and other samples with 0.5 mass%, 1.0 mass%, 2.0 mass% of nano-SiO_2 in paraffin wax, respectively. Similarly, the mass% of residues remained after the degradation was obtained as 0%, 0.2%, 0.6%, and 0.9%, respectively. Paraffin wax was removed at 260°C, and all other samples were completely degraded at 450°C. The result ascertained that the dispersion of SiO_2 nanoparticles expressively delayed the thermal decom- position of base paraffin wax as reported by Rao et al. (2014) and Karaipekli et al. (2017).

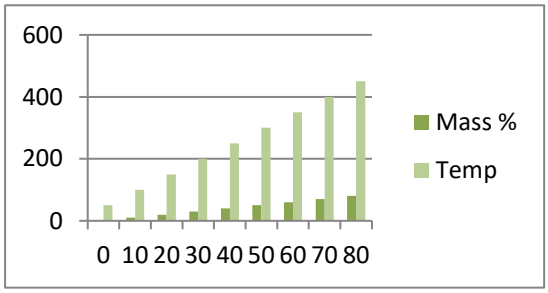

Figure 6. TGA results of nano-SiO2/paraffin PCM samples.

Thermal conductivity measurement

Thermal conductivity of the nano-enhanced PCMs is purely depending on the degree of dispersion of the nanoparticles in the base PCM. The dispersion is contingent on the duration of stirring and ultrasonication of the mixture. The effective ultrasonication ensures the finest suspension of nano-particles in the PCM, which will form the decent network to transfer heat to all the portion of the PCM rapidly (Jesumathy, Udayakumar, and Suresh 2012). The thermal conductivity of the samples, measured through the experimentation is presented in Figure 7. Thermal conductivity of the pure paraffin is 0.18, and it has been increased to 0.203, 0.221, and 0.24 for the samples with 0.5 mass%, and 2.0 mass% of nano SiO_2 in paraffin wax, respectively. Ji et al. (2012) acknowledged that the dispersion of multi-walled carbon nanotubes in palmitic acid increased thermal conductivity to 67%. Deng et al. (2016) concluded that the addition of silver nanowire in polyethylene glycol PCM scrupulously improved the thermal conductivity to 1130%, which is 11.3 times higher than the thermal conductivity of pure polyethylene glycol. The percentage enhancement in thermal conductivity (k) of nano-SiO_2/paraffin PCM was calculated as per the following equation,The percentage enhancement in thermal conductivity of nano-SiO_2/paraffin PCMs found to be 12.78%, 22.78%, and 33.34%, respectively, compared to the pure paraffin wax as in Figure 8. Relatively, the percentage improvement in thermal conductivity of paraffin wax is not impressive at 2.0 mass% of SiO_2 nano particles comparing to other lower mass%. Furthermore, the result shows that the thermal conductivity is increasing non-linearly with the mass% of nano particles. Mohamed et al. (2017) obtained the same pattern of result, when they conducted an experiment with paraffin wax and α nano alumina. The reason behind the non-linear variation of thermal conductivity is due to the

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uneven dispersion and random agglomeration of nanoparticles, when the mass% of nano- particles is increased.

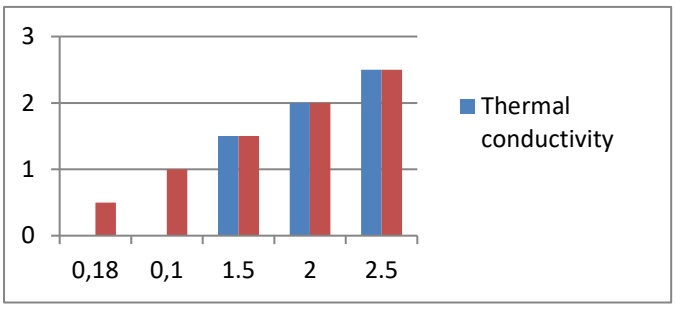

Figure 7.Variation in thermal conductivity for different mass% of nano-SiO2/paraffin PCM samples.

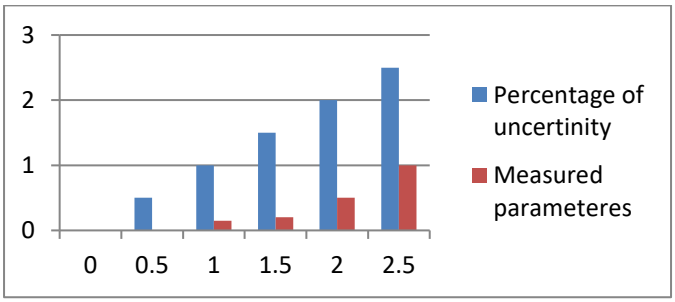

Figure 8.Variation in thermal conductivity percentage for different mass% of nano-SiO2/paraffin PCM samples.

Table: 4 Measuring Parameters

S.No.	Measuring parameter	Instrument	Model	Possible error	Uncertainty
1	Particle Size	Particle Size Analyzer	Zetasizer AT	±0.006 nm	±2%
2	Chemical Stability	FT-IR Spectrometer	PerkinElmer Spectrum Two	±0.1 cm^{-1}	±0.0025%
3	Latent Heat	DSC	DSC 6000 with Pyris 6	±2.8 kJ/kg	±2%
4	Melting and Solidification Point	DSC	DSC 6000 with Pyris 6	±0.1°C	±0.15%
5	Degradation Temperature	Thermo gravimetric Analyzer	TGA 4000	±1°C	±0.2%
6	Thermal Conductivity	Thermal properties analyzer	KD2 Pro	±0.01 W/mK	±0.5%

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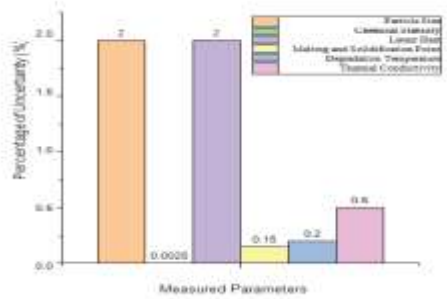

Figure 9.Uncertainties of various measured parameters.

Uncertainty analysis

The results attained from different measuring instruments may be ended up with uncertainties. These uncertainties ould be arisen due to the factors such as errors in calibration of the measuring instruments and accuracy of the instruments used. In order to ensure the correctness of the obtained result, a detailed uncertainty analysis was conducted using the mathematical expression proposed by Naghavi et al. (Magahi et al. 2017) as given below:

The calculated uncertainties for various measured parameters are presented in Table 4. The graph showing relative uncertainties of various measured parameters is portrayed in Figure 9.

Conclusions

In this work, the nano-enhanced phase change materials had been prepared by dispersing different mass% of nano-SiO_2 particles in paraffin wax and their thermal properties were investigated to make use of them as thermal energy storage medium in solar heating application. The four samples; 0 mass%, 0.5 mass%, 1.0 mass%, and 2.0 mass% of nano-SiO_2 in paraffin wax had been prepared using two-step method. Each. sample was experimentally investigated by means of different experimental techniques such as field emission scanning electron microscope (FESEM), differential scanning calorimetry (DSC), thermogravi- metric analysis (TGA), Fourier transform infrared spectroscopy (FT-IR) and thermal conductivity test. The results are concluded as follows:

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[13]. The scanning electron microscope (SEM) analysis ensured the distribution of nano-SiO_2 particles in paraffin wax and formation homogenous mixture of nano-SiO_2/paraffin PCMs.

[14]. The differential scanning calorimetric (DSC) results showed that the melting temperature of the nano-SiO_2/paraffin PCMs were reduced to 63.10°C, 62.72°C, and 62.08°C, respectively, and solidification temperature enhanced to 57.15°C, 57.54°C, and 59.41°C, respectively. This phenomenon ensures that the nano-SiO_2 particles reduced the super cooling of the paraffin wax. On the other hand, the latent heat of the paraffin wax was reduced with the increased mass% of nano-SiO_2 particles and the declination was maximum at 2.0 mass% of nano-SiO_2 particles.

[15]. The Fourier transform infrared (FT-IR) results proved that the physical bonding strongly exists between the paraffin wax and nano-SiO_2 particles. Also, it confirmed that the chemical structure of the paraffin wax is not affected by the addition of nano-SiO_2 particles.

[16]. Thermo gravimetric analysis revealed that the incorporation of nano-SiO_2 particles in paraffin wax increased the thermal degradation temperature of the paraffin wax; thereby it delayed the decomposition of paraffin wax to the high temperature.

[17]. Thermal conductivity test exhibited that the thermal conductivity of the paraffin wax was enhanced to 12.78%, 22.78%, and 33.34%, respectively, for the samples with 0.5 mass%, 1.0 mass%, and 2.0 mass% of nano-SiO_2 particles and at 2.0 mass%, the improvement was not perceptible compared to other samples with lower mass% of nano-SiO_2 particles.

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