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DETERMINATION OF THERMAL PROPERTIES OF A82 ORGANIC PHASE CHANGE MATERIAL EMBEDDED WITH DIFFERENT TYPE NANOPARTICLES

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Abstract: The low thermal conductivities of organic phase-change materials must be increased so that they can be used effectively in practice. In this work, it is aimed to enhance the thermal conductivity of PCM by adding nanoparticles into PCM. The effect of used nanoparticle type, size and shape on the thermal properties such as thermal conductivity, latent heat, melting / solidification temperatures has been systematically investigated. The thermal conductivity enhancement of composites doped with carbon based nanoparticle were found to be higher than that of the composites doped with metal oxide nanoparticles. 154.9% improvement was achieved in the thermal conductivity of the phase change material composite doped with 5% GNP. It has been observed that there is no significant change in melting/ solidification temperatures (<1.5 °C) depending on the doped metal oxide or carbon-based nanoparticles, but deterioration up to 20% in latent heat. However, ZnO nanoparticles have been found to have the effect of enhancing latent heats. In addition to the improvements achieved in the thermal conductivities of phase change materials, their stability are protected over the long term. This result show that it can be used as an energy storage material.
Keywords: Nanoparticle, Thermal conductivity, Phase change material, Differential scanning calorimetry

FARKLI TİPTE NANOPARÇACIKLARLA KATKILANAN A82 ORGANİK FAZ DEĞİŞTİREN MALZEMENİN TERMAL ÖZELLİKLERİNİN BELİRLENMESİ

Özet: Yüksek enerji depolama kapasitesine sahip organik faz değiştiren malzemelerin uygulamada etkin bir biçimde kullanılabilmeleri için düşük ısıl iletkenliklerinin iyileştirilmesi gerekmektedir. Bu çalışmada organik bir faz değiştiren malzeme içerisine nanoparçacıklar katkılanarak ısıl iletkenliğin iyileştirilmesi amaçlanmıştır. Kullanılan nanoparçacıkların tip, boyut ve şeklinin, termal özellikler (ısıl iletkenlik, gizli ısılar, erime/katılaşma sıcaklıkları) üzerindeki etkisi sistematik olarak incelenmiştir. Karbon tabanlı nanoparçacık katkılanan organik faz değiştiren malzemelerin ısıl iletkenliklerinde görülen iyileşmenin metal tabanlı nanoparçacık katkılanan faz değiştiren malzemelere göre önemli derecede daha yüksek olduğu belirlenmiştir. %5 GNP katkılanan faz değiştiren malzeme kompozitinin ısıl iletkenliğinde %154.9 iyileşme sağlanmıştır. Katkılanan metal oksit veya karbon tabanlı nanoparçacık bölüntüsüne bağlı olarak erime/katılaşma sıcaklıklarında önemli bir değişim olmadığı (<1.5 °C) buna karşın gizli ısılarda %20'ye varan bir azalma olduğu gözlemlenmiştir. Buna karşın ZnO nanoparçacıkların gizli ısıları iyileştirmek gibi bir etkisinin olduğu tespit edilmiştir. GNP katkılanan faz değiştiren malzemelerin ısıl iletkenliklerinde elde edilen iyileşmelerin yanı sıra uzun dönem boyunca kararlılıklarını koruyabilmesi onun bir enerji depolama malzemesi olarak kullanılabileceğini göstermiştir.
Anahtar kelimeler: Nanoparçaçık, Isıl iletkenlik, Faz değiştiren malzeme, Diferansiyel taramalı kalorimetre

LIST OF SEYMBOLS

D	Diameter [m]
GNP	Graphene Nano Platelet
H	Latent heat [J/g]
m	Mass [g]
MWCNTs	Multi Walled Carbon Nanotubes
L	Length [m]
PCM	Phase Change Material
T	Temperature [°C]
x	nanoparticle mass fraction [%]

Subscripts

em	endset melting
m	melting
np	nanoparticle
om	onset melting
s	solidification
es	endset solidification
os	onset solidification

INTRODUCTION

The phase change materials (PCMs) are known as energy storage material that capable of phase change transition from solid to liquid or liquid to solid at small temperature range and possess high energy storage capacity (Sarier and Onder, 2102). Because of their high thermal storage capacities, PCMs have a potential to be used in a variety of engineering fields such as space, defense, automotive, textile and construction. Recently, researchers have been focused on the use of PCMs in many applications such as thermal energy storage (Farid et al., 2004, Dincer et al., 2002, Zalba et al., 2003), thermal protection (Garimella 2006, Cao et al., 1990, Ho et al., 1996) and active and passive cooling of electronic devices (Bellettre et al., 1997, Pal et al., 1997, Pincemin et al., 2008). In our previous work (Parlak et al., 2016) thermal performance of GNP/PCM composite was investigated in an energy storage unit that could be used for thermal protection of temporary working electronic equipments. It has been found that the heat received in energy storage unit is more efficiently spread in the energy storage unit due to the increase of the GNP fraction added into the PCM.

The different types of solid-liquid PCMs such as organic (paraffin) and inorganic (hyrdrate salt) PCMs can be used widely at industrial applications. However the organic PCMs are the most preferred because of having some advantages such as high latent heat, low vapor pressure, chemical and thermal stability and easily and economically attainable (Sharma et al., 2009). In contrast, organic PCMs have a very low thermal conductivity which significantly restricts the efficient use of PCMs in practice. Therefore, it is a good strategy to incorporate materials with high conductivity into organic PCMs to enhance the thermal properties of PCMs for engineering applications. For this purpose, various techniques such as inserting high conductivity fins (Agyenim et al., 2009, Zhang et al., 2015), foams (Xiao et al., 2002) and matrices (Yang et al., 2016) have been implemented from past to date. Although these methods improve the thermal conductivities of PCMs, they have disadvantages such as increasing system weight and volume, having poor stability, and reducing the amount of PCM used (Wang et al., 2010).

Over the last decade, the synthesis of nanomaterials in different sizes (<100 nm) and types led to development of a more innovative method for increasing the thermal conductivity of organic PCMs. This method is based on the formation of a new nanoparticle/PCM composite by incorporating nanoparticles into the organic PCM in certain amounts (Kumerasan et al., 2012). Several studies have been carried out to investigate effect of the loading of nanoparticles into the PCMs for enhancement of thermal conductivity. In the literature both metal/metal oxide (Wang et al., 2010, Ho et al., 2009, Wu et al., 2010) and carbon based nanoparticles (Cui et al., 2011, Zeng et all 2008, Wang et al., 2009, Wang et al., 2010, Kumerasan et al., 2012, Wang et al., 2016) have been used to improve the thermal conductivity of PCMs. In general, studies in literature, on improving the thermal conductivity of organic PCMs by loading nanoparticle show that different improvement ratios are obtained in thermal conductivity despite the same type of nanoparticle doping. For example, Wang et al., 2010 achieved 26% improvement in thermal conductivity by doping with mass fraction of 5% Al_2O_3 nanoparticles in organic PCM. In contrast, Ho et al., 2009 achieved only 17% improvement in thermal conductivity, despite of 10% mass fraction of Al_2O_3 nanoparticle doping. Similar situations are also mentioned in the thermal conductivity improvements of carbon-based nanoparticle doped organic PCMs. Ciu et al., 2011 achieved 24% improvement in thermal conductivity by adding MWCNTs in a 5% mass fraction into organic PCM, while Wang and et al.,2010b achieved 51.6% improvement by adding 1% MWCNTs to the mass. The reason why the improvement in thermal conductivity is different despite same type of nanoparticle doping can be explained as follows: the use of different organic PCMs, the use of nanoparticles of different sizes and pre-treatment of nanoparticles before doping. In the literature, the number of studies to compare nanoparticles effects on improving PCM thermal properties is rather limited. Moreover, in these limited studies despite the comparison of different types of nanoparticles, no attention was paid to the nanoparticle size. As can be seen from the brief literature review, there is a lack of systematic investigation to compare the thermal conductivity enhancement of nanoparticles incorporated in organic PCMs.

Therefore, this study focuses on examining the thermal properties of nanoparticle / PCM composites prepared by applying the same method and using different nanoparticles. In accordance with this purpose, firstly the systematical investigation about the effect of the different nanoparticles of the same size on the thermal properties of the PCM were carried out. Then the size effect of the same nanoparticle on thermal properties of the PCM were examined detailed.Thermal conductivity and DSC property measurements were performed to determine the thermal properties of PCMs doped with nanoparticles such as Al_2O_3, TiO_2, MgO, ZnO, MWCNTs and Graphene nanoplatelet (GNP) in fractions of 1% to 5% by mass.

MATERIAL AND METHOD

Paraffin wax organic PCM (A82) with a melting temperature of 82 ^oC was commercially supplied from Phase Change Materials Products Limited (United Kingdom) and used without any pretreatment. Metal oxides, MWCNTs and GNP nanoparticles were commercially supplied from US Research Nanomaterials Inc (USA) and Skyspring Nanomaterials Inc (USA) respectively.

The mass fraction of nanoparticles to be doped into the PCM was calculated using Equation 1.

$$\frac{x}{100} = \frac{m_{np}}{m_{np}+m_{\mathrm{A82}}} \tag{1}$$

where; x is the nanoparticles mass fraction (%), m_{np} is the amount of nanoparticles (gr) and m_{A82} is the amount of PCM (gr).

Nanoparticles/A82 composites were prepared by appliying two steps melting-mixing procedure. In the first step, the PCM was melted by heating to its melting temperature on a heating plate. Then 1%, 3% and 5% mass fraction of nanoparticles were doped into the liquid A82. In the second step, in order to provide uniformly dispersion of nanoparticles into liquid A82, stirring process was carried out by using an 750 W ultrasonic stirrer (Sonics&Materials INC, USA) for 30 minutes. The temperature of mixture was kept above the A82 melting temperature in order prevent composite to solidify during mixing process.

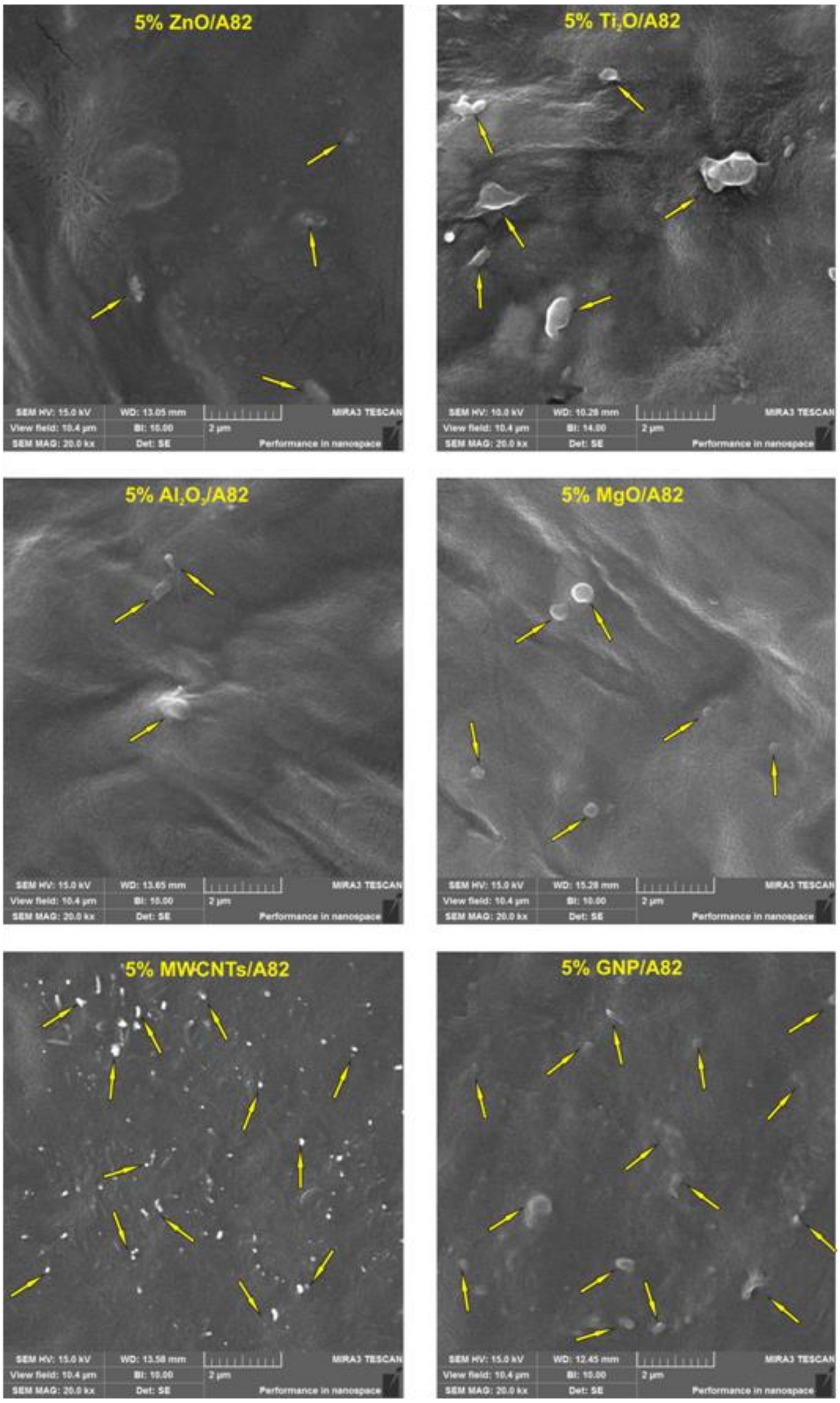

Figure 1. SEM images for dispersion capability of the nanoparticles within the PCM

In order to evaulate dispersion capability of the nanoparticles within the PCM matrix the SEM (Tescan Mira 3 XMU, Czechia) images of the composite that doped with different types of nanoparticles at 5% was given in Fig. 1. Because nanoparticles are heavier than PCM, they appear brighter in image due to more efficient back scattering of electrons. It can be seen from Figure1 that the metal nanoparticles were exposed to strong agglomeration due to the high density and were not uniformly dispersed within the PCM On the other hand, carbon-based nanoparticles were more uniformly dispersed in PCM than metal oxide nanoparticles. According to SEM images, it can be said that carbon-based nanoparticles were formed a more effective structure in terms of heat conduction.

In order to obtain solid measurement samples to be used in measurement of thermal conductivity, melted nanoparticle/A82 composite was poured into the acrylic cylindrical mold with 30 mm in diameter and 150 mm in height. The shaft on the cylinder mold axis allowed manufacturing of the samples with holes of 2.4 mm on to provide sensor entry. After solidification of composites were provided at room temperature, test samples removed from the mold and used for thermal conductivity measurement.

The thermal conductivities of nanoparticle/A82 composites were performed using the KD2 Pro device (Decagon Devices Inc, USA). The KD2 Pro operates on the basis of a transient linear heat source and it consists of a microcontroller unit and a sensor with a diameter of 2.4 mm in diameter and length of 100 mm. The high L/D ratio ensures that the tip effects are neglected. The sensor operates both as a heater and a temperature sensor and determines the temperature change during the flow of current through the heater for a certain time period. The conductivity measurements for all samples were performed at laboratory temperature. It is expected to provide thermal equilibrium between the samples and the environment before starting the measurements. Five measurements were performed for each sample and the mean values with a 0.5% standard deviation were given as a result. The accuracy of the measurements ±10% for 0.2-4 W/mK range and ±0.02 W/mK for 0.1-0.2 W/mK range. Other thermal properties, such as melting/solidification temperatures and latent heats, were measured using a Differential Scanning Calorimeter (DSC) (Shimadzu Corporation, Japan). DSC samples were prepared using powdered particles in about 5 mg from the nanoparticle/A82 composites produced. The electronic scale (Shimadzu Corporation, Japan) with 0.01 mg precision was used for weighing of the powdered composites. The DSC samples were obtained as a result of the powdered composites being placed and pressed together with a lid in a special aluminum. The calibration of the DSC instrument was performed using standard indium sample with high purity. DSC measurements were performed a heating/cooling rate of 2 °C/min for a range of 30 °C-120 °C. Three measurements were made for each sample and the mean values with a 1% standard deviation were given as a result. The calorimetric sensitivity and temperature accuracy of the DSC are ±1% and 0.1 °C respectively.

Thermal performance of composites were obtained by measuring variation of temperatures over time at a single point within the energy storage unit. All the composites were placed in a 40x40x40 mm cube that was made of castermid material with low thermal conductivity (0.29 W/mK) (Figure 2a). The walls of the energy storage unit are in sufficient thick in order to allow for long-term usage at high temperatures. The energy storage unit was heated from the bottom by means of plate type resistance. A cooling plate was placed to the top of the unit to allow for heat transfer to go vertically upwards. Measurements were made at a middle point of the module by using J type thermocouples. Thermocouples were placed in the energy storage unit by using thermocouples connection element that was made of teflon material. A DC power supply is directly connected to the resistance to provide 25 W heat load. Temperatures values were taken every 30 seconds by means of a data logger and saved on the computer (Figure 2b).

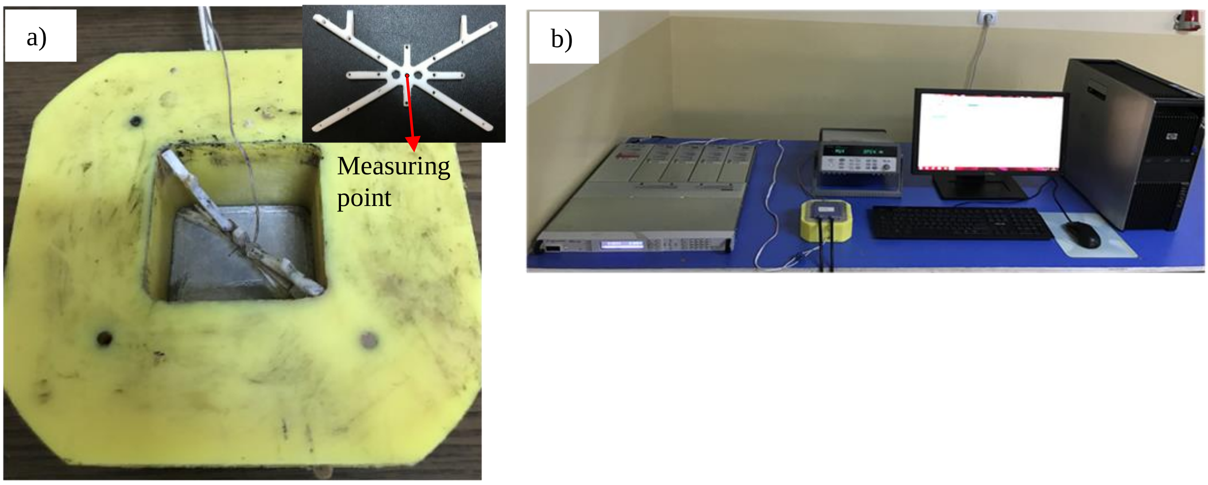

Figure 2. The experimental set up for the thermal performance measurements

RESULTS AND DISCUSSIONS

The variation of thermal conductivities of the nanoparticle/A82 composites according to mass fraction of nanoparticles is given in Figure 3. First of all, the thermal conductivity coefficient of A82 is measured as 0.308 W/mK. The thermal conductivity of A82 is low due to both low frequency of vibration and inhibition of phonon transfer. In contrast embedding nanoparticles that have high vibration frequency, into A82 leads to improved phonon scattering and therefore resulting in enhanced heat conduction.

It is seen from the figure that there is an increase in thermal conductivities of composites (except 1% Al_2O_3/A82) with the increase of nanoparticle mass fractions. The enhancement in the thermal conductivities of A82 composites doped with metal oxide nanoparticles were found to be quite low compared to carbon based nanoparticles.The enhancement in thermal conductivity of A82 composites doped with 5% mass fractions of ZnO(18nm), TiO_2(10-25nm), Al_2O_3(20nm) and MgO(20nm) nanoparticles were measured as 2.6%, 3.6%, 6.5% and 8.4 respectively. In contrast, enhancement in the thermal conductivity of 5% MWCNTs/A82 and 5% GNP/A82 composites were measured as 26.7% and 154.9%, respectively.

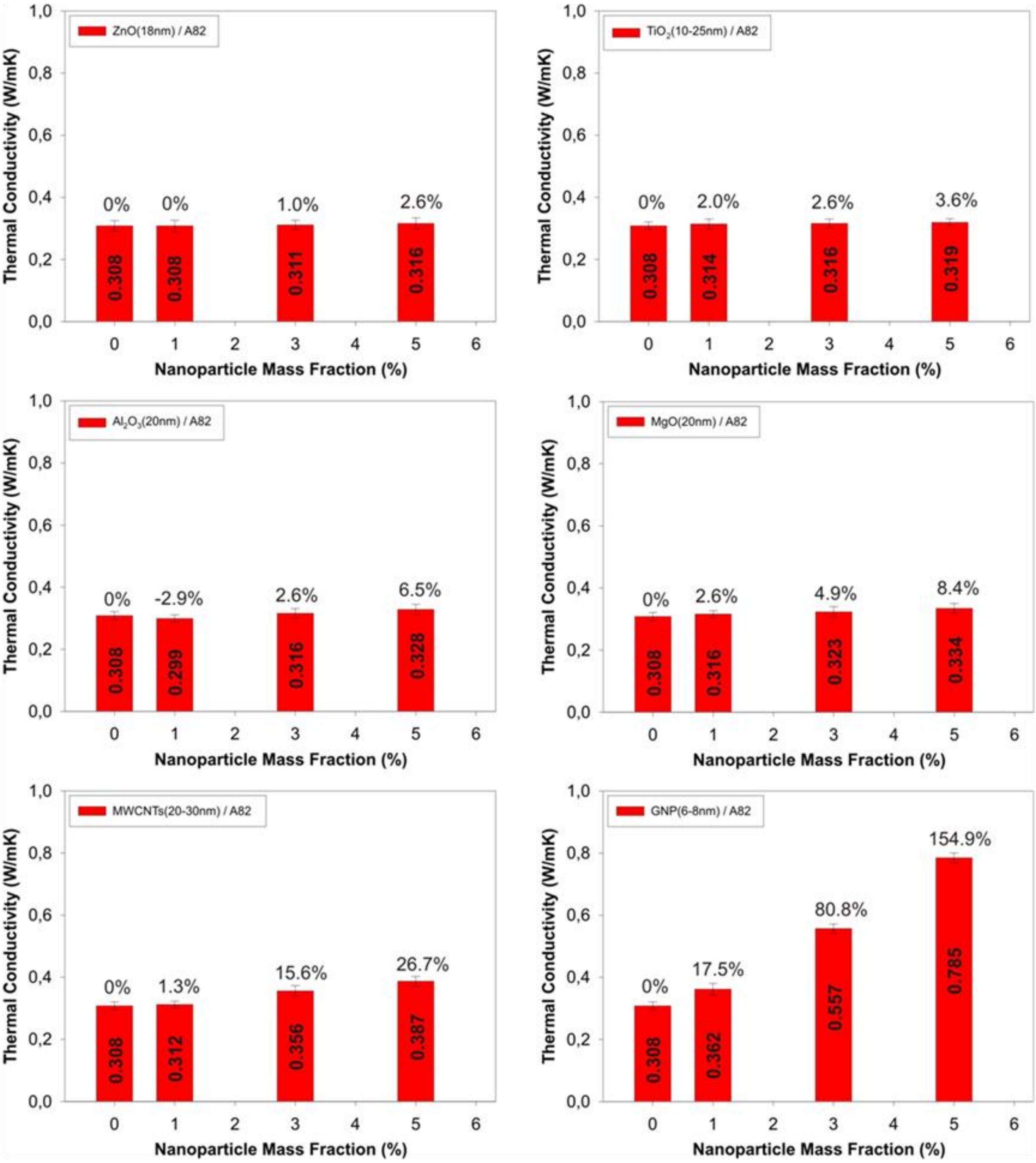

Figure 3. The variation of thermal conductivities of the nanoparticles/A82 composites

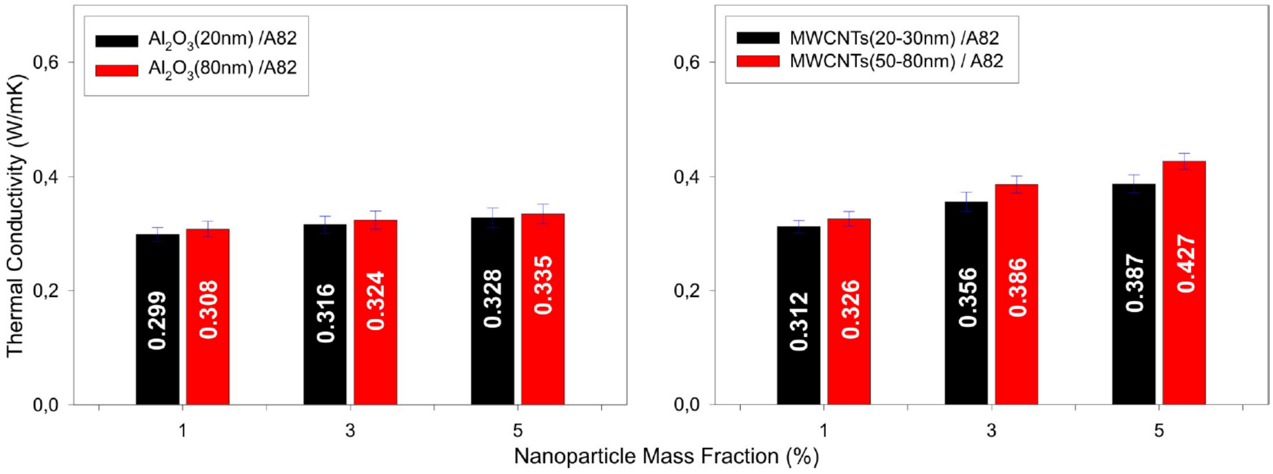

Figure 4. Size effect of nanoparticles on thermal conductivity

The reasons for the different improvement in the thermal conductivity of composites that doped with different nanoparticles having about same dimensions can be summarized as follow: i) The thermal conductivities of nanoparticles are different. Especially carbon-based nanoparticles have higher thermal conductivities (>3000 W/mK at planar directions) than metal oxide nanoparticles, ii) The nanoparticles shape have significant effects on the thermal conductivity enhancement. If the nanoparticles used in the study are evaluated geometrically, it can be said that the metal oxide nanoparticles are spherical, the MWCNTs nanoparticles are in the tube shapes, and the GNP nanoparticles are in the two-dimensional planar structure. It can be said that the MWCNTs and GNP, which have essentially the same thermal conductivity value, cause different enhancing in thermal conductivity within the A82 depending on the different nanoparticle shape. In this case, it can be thought that nanoparticles with high surface/volume ratio form a network that allows better phonon scattering in the A82.

The size effects of nanoparticles on the thermal conductivity enhancement were carried out both for Al_2O_3 and MWCNTs nanoparticles. Figure 4 shows the variation of thermal conductivity of the composites that doped with nanoparticles at different sizes depending on the nanoparticle mass fractions. It has been found that the increase in nanoparticle diameter in both types of nanoparticles has a positive effect on the improvement in thermal conductivity. This is the result of a decrease in resistance in the nanoparticle-nanoparticle and nanoparticle-PCM interfaces due to increased surface area.

Long-term stability of thermally enhanced composites is a prerequisite for their reliable using in the energy storage applications. The reliability of the composites can be determined by demonstrating that the thermal conductivities are stable. Figure 5 shows the thermal conductivity of the 5% GNP/A82 composites after they have undergone 5, 10,15 and 20 heating/cooling cycles. As can be seen from the figure, the maximum variation in thermal conductivity less than 2.5% over the performed heating/cooling cycles. This indicates that GNP nanoparticles did not undergo significant phase separation within A82. This results shows that GNP/A82 composites are highly stable and can be used in energy storage applications.

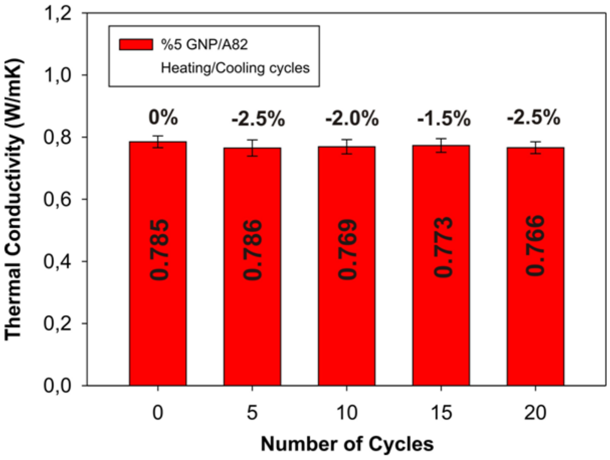

Figure 5. Heating/cooling cycles effect on thermal conductivity

The thermogram curves for the A82 and other composites were obtained as a result of DSC analysis composed of melting and solidification and given in Figure 6. Here, the curves obtained during the melting and solidification process are called the endotherm and exotherm curve respectively. The peaks formed by the exotherm and endotherm curves show the part related to the phase change. By analyzing the endotherm curves; melting onset temperatures (T_{om}), melting endset temperatures (T_{em}) and melting latent heats (H_m) were obtained. Solidification onset temperatures (T_{os}), and solidification end temperatures (T_{es}) and also solidification latent heats (H_s) were similarly obtained by the analysis of exotherm curves.

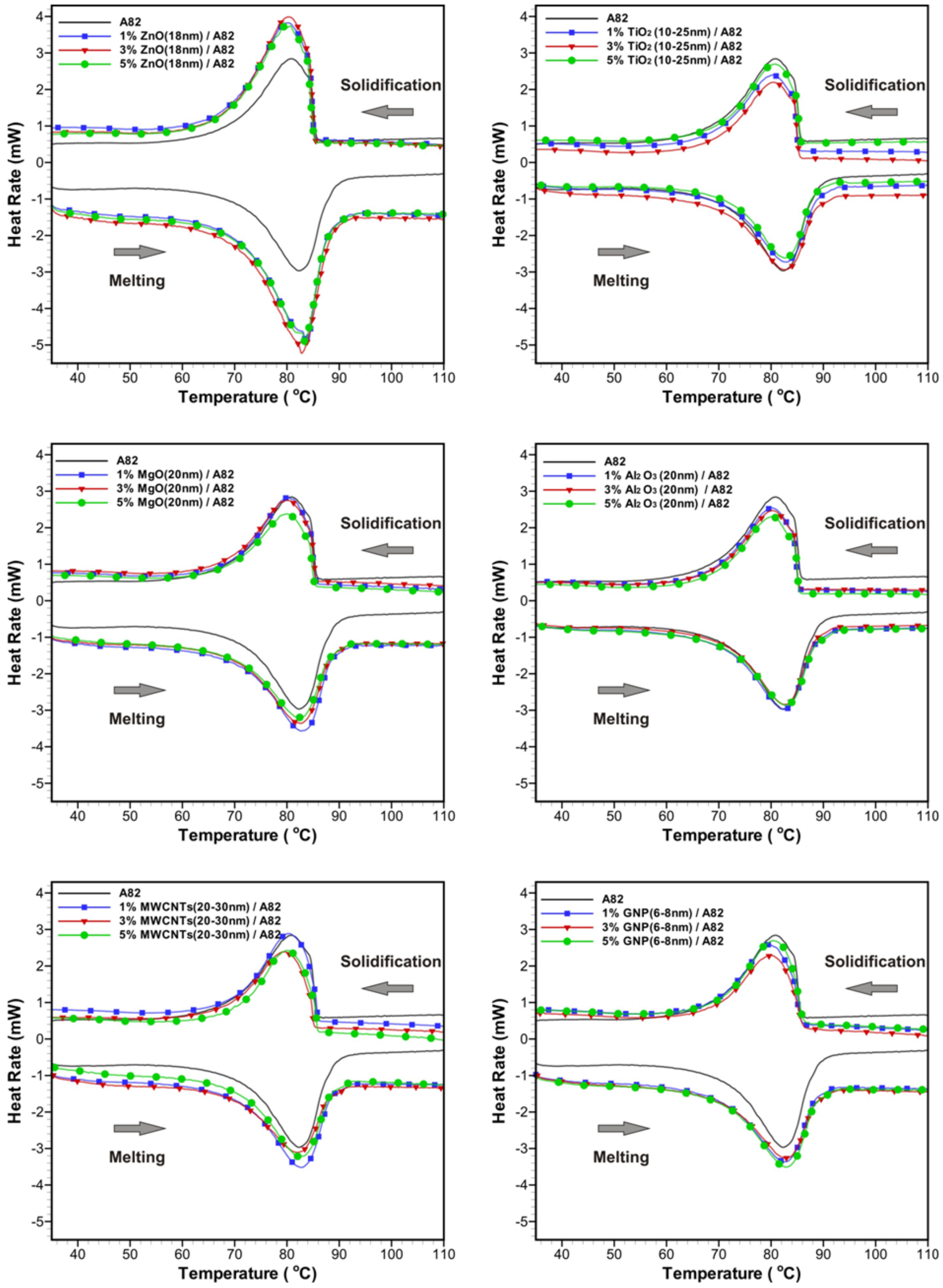

Figure 6. The thermogram curves of nanoparticles/A82 composites

Firstly, exotherm and endotherm curves for the A82 were analyzed and their DSC properties were determined as follows. The melting DSC properties were obtained by analysing the endotherm curve. The solidification/melting phase change of A82 starts at 70.8 ^{o}C (onset melting temperature) and ends at 88.7 ^{o}C (endset melting temperature). The area under the endotherm peak is a measure of melting latent heat (H_m) and measured as 175.6 J/g. Similarly the solidification DSC properties were obtained by analysing the exhotherm curve. The melting/solidification phase change of A82 starts at 86.3 ^{0}C and ends at 68.9 ^{o}C respectively. The solidification latent heat (H_s) was measured as 159.4 J/g from the area under the exotherm curve. The thermogram curves of the composites doped with metal oxide and carbon-based nanoparticles at mass fraction range of 1% - 5% were found to be quite similar to the clean A82. Accordingly, it has been found that there is no significant change in the melting and solidification temperatures of all composites depending on the nanoparticle types and mass fractions.

Table 1. DSC properties of nanoparticles/A82 composites (for 5% mass fractions)

Composites	T_{om} (ºC)	T_{em} (ºC)	H_m (J/g)	T_{os} (ºC)	T_{es} (ºC)	H_s (J/g)
A82	70.8	88.7	175.6	86.3	68.9	159.4
5% ZnO(18nm)/A82	70.6	88.3	234.8	85.7	67.9	229.1
5% TiO_2(10-25nm)/A82	71.0	88.7	144.6	86.3	69.7	146.5
5% Al_2O_3 (20nm)/A82	70.5	89.1	155.6	86.1	68.5	147.7
5% MgO(20nm)/A82	70.0	88.8	154.6	85.9	68.2	138.9
5% MWCNTs(20-30nm)/A82	70.3	89.0	151.0	86.3	69.6	148.5
5% GNP(6-8nm)/A82	70.9	89.1	155.0	86.4	70.0	150.1

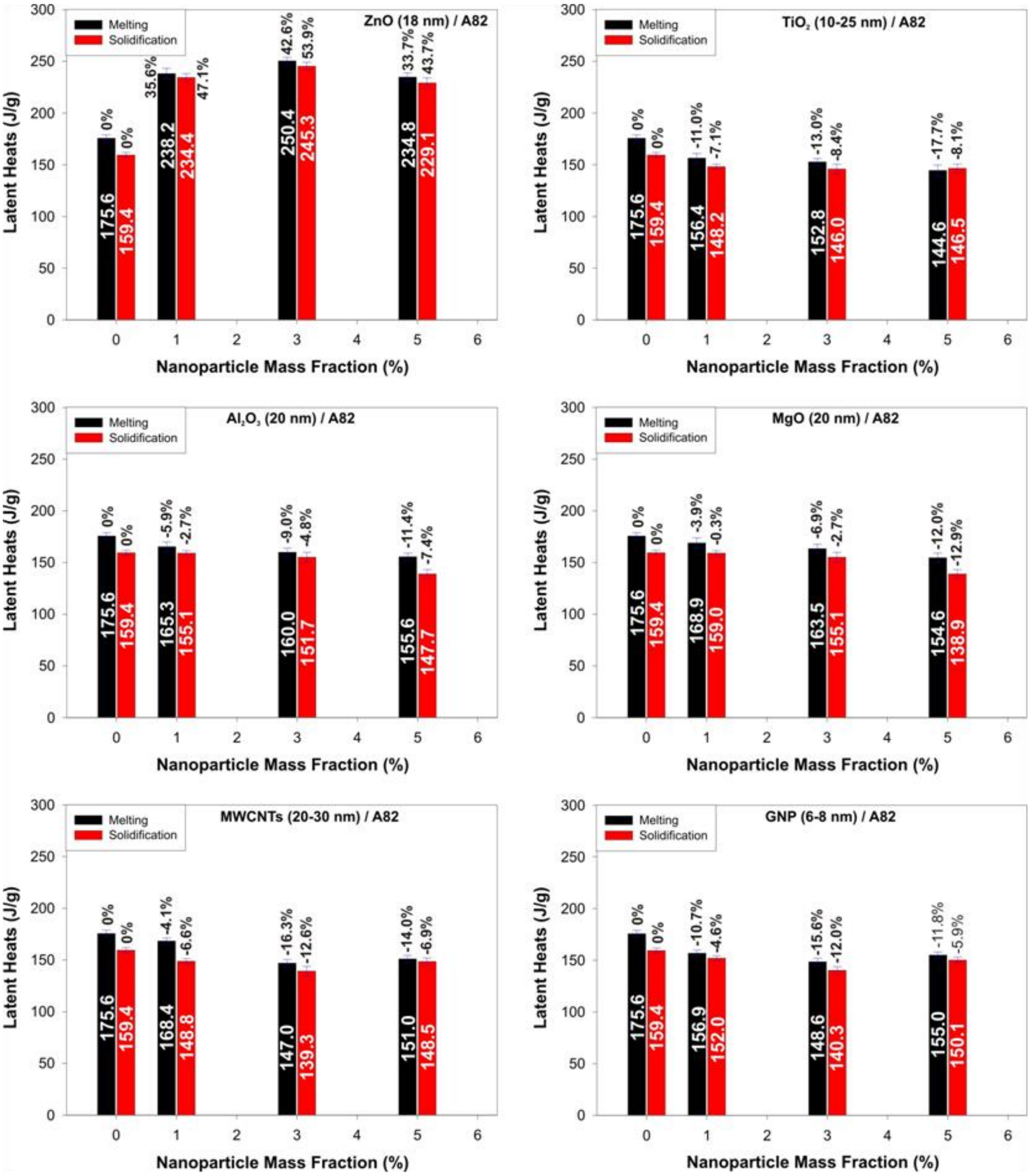

Figure 7. The variation of melting and solidificitaion latent heats with respect to the doped nanoparticle mass fractions

Table 1 demonstrates the DSC properties of nanoparticle-doped composites at 5% by mass. As can be seen from the table that the maximum variation in temperatures less than 1.5%. Similar results are valid for composites doped with nanoparticles in other mass fractions. However in general the areas under the endotherm and exotherm curve decrease (except ZnO/A82 composites) as the nanoparticles mass fraction increases. In other words, certain decrease in the melting/ solidification latent heats were occured depend on the increase of the nanoparticle mass fraction within the A82.

As mentioned above the melting and solidification latent heats of A82 were measured as 175.6 J/g and 159.4 J/g respectively. As will be noted, the solidification latent heat is smaller than melting latent heat due to the incomplete crystallization (Fan et al., 2013). The variation of melting and solidificitaion latent heats with repect to doped nanoparticle mass fraction were given in Figure 7. It was determined that latent heats of all composites except for the composites doped with ZnO nanoparticles decreased. Nanoparticles have low energy storage capabilities despite high thermal conductivities. It is known that the energy storage property deteriorates if a material having good energy storage property is mixed with a material having poor energy storage property according to mixing law. Depending on the increase of nanoparticle mass fractions, deteriorations in latent heats were determined in the range of 5%-20%. Conversely, the latent heats of composites doped with ZnO nanoparticles exhibit an improvement of 30% to 55%. The thermal energy storage property of materials is related to strong ionic and covalent bonds between molecules. The intermolecular attraction between the molecules of nanoparticles and organic PCM was considered as the possible reason for the enhancement of latent heat values (Shaikh et al., 2008). In this case, it can be said that ZnO nanoparticles increase intermolecular attractions.

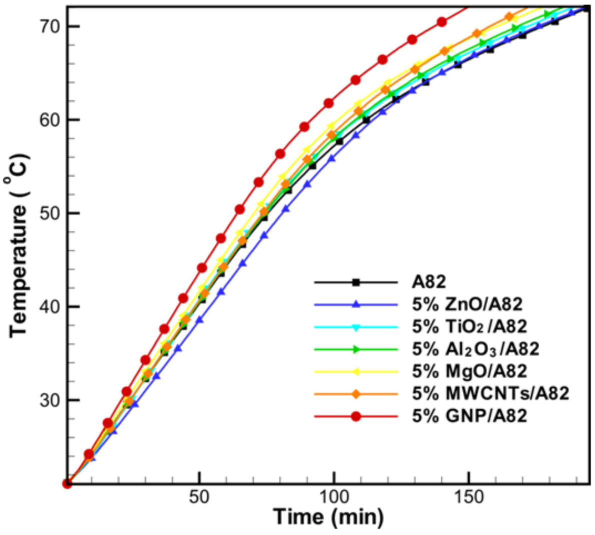

Figure 8. Time dependent temperature variations of nanoparticles/A82 composites.

Figure 8 shows the variation over time regarding temperature values at a middle point of the composites that is heated by means of plate type resistance from the bottom. It was seen that time dependent increases in the temperature were slowed down at high temperatures (>50 ºC) for all composites. This is an indication that the composite thermal conductivity decreases with increasing temperature. It is clear that thermal responses of the A82 and nanoparticle/A82 composites differ significantly. It has been observed that the time-dependent temperature increase of 5% nanoparticle-doped composites increases in parallel with the improvement in thermal conductivity.

The thermal performances of the nanoparticles/A82 composites were determined by the comparison of the times at which the measurement point temperature reached 72 ºC. Table 2 gives the time values of the A82 and nanoparticles/A82 composites. The time to reach the measurement point temperature of 72 ºC was measured as 195 minutes for A82. It was determined that time values were shortened depend on the improvement in thermal conductivity. As expected the best thermal response have been seen in GNP/A82 composites that have high thermal conductivity. It was said that the thermal performance of A82 was improved as 23.6% by doping 5% GNP nanoparticles.

Table 2. Comparison of reach times to 72ºC

Composites	**Time (min)**
A82	195
5% ZnO(18nm) / A82	193
5% TiO_2(10-25nm) / A82	189
5% Al_2O_3(20nm) / A82	185
5% MgO(20nm) / A82	177
5% MWCNTs(20-30nm) / A82	171
5% GNP(6-8nm) / A82	149

CONCLUSIONS

In this work, the thermal properties of nanoparticle/A82 composites doped with carbon or metal based nanoparticles have been investigated in detail. Both thermal conductivity and DSC measurements were performed to determine the thermal properties. The results obtained are given below.

The enhancement in thermal conductivity of composites doped with metal oxide nanoparticles were measured below 10%.

The enhancement in thermal conductivities of composites doped with carbon-based nanoparticles is significantly higher than that of the composites doped with metal oxide nanoparticles. Thermal conductivity enhancement of MWCNTs/PCM and GNP/PCM are measured as 26.7% and 154.9% respectively.

It was determined that maximum variation in the melting/solidification temperatures of all composites less than 1.5% depending on the doped nanoparticles.

In general, up to 20% decreases in latent heats have been observed due to the increase in the nanoparticle mass fraction added. On the contrary, the latent heats of ZnO/A82 composites showed improvement in the range of 30-55%.

As the temperature increases, the thermal conductivity of all composites decreases.

The thermal performance of A82 is improved as 23.6% by doping 5% GNP nanoparticles.

The better long-term stability of GNP/A82 composites has shown that homogeneous distribution of nanoparticles within the PCM is maintained. This result demonstrates that GNP/A82 composites can be used for energy storage purposes.

In the future study, it is aimed to prepare GNP/ZnO/PCM composites to improve both thermal conductivity and latent heats.

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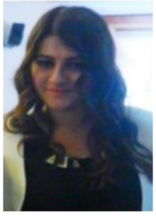

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