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Research Article

Investigation of glycerol-Ni(NO₃)₂·6H₂O /perlite composites as form stable phase change materials

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Abstract

In this presented experimental research, composites of phase change materials (PCMs) were prepared by impregnating glycerol and nickel salt into the expanded perlite pores (EP). In this study, vacuum impregnation and wet impregnation methods were used. Glycerol and nickel salt were inserted to the pores of EP and during the phase transition the leakage of the glycerol and nickel salt were prevented. The characterization study of chemical structure and surface microstructure of the composite PCMs were performed by Fourier transformation infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). Thermal properties were determined by differential scanning calorimeter (DSC) instrument. The melting temperature and latent heat were determined for prepared composite, as -8.3°C and 136 J/g for vacuum impregnation method. For wet impregnation method the melting point could not be measured because of the PCM mixture could not deposited into perlite by using this method. The FTIR analysis presented that no chemical reactions take place between the EP and prepared PCM mixture. SEM images showed that the glycerol nickel salt mixture dispersed uniformly into the EP pores. During the heating and cooling processes, leakage and impairment of the composite PCM were not detect. Consequently, the PCM composite obtained by vacuum impregnation method, can be used for thermal energy storage applications.

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1. Introduction

Industrial development and rising population increase the energy consumption in the world. It is a known fact that the primary used energy resources of the world are going to exhausted rapidly and also the usage of traditional energy sources are main cause of the environmental pollution. Energy storage seems as a good opportunity to solve the energy problem in the world as well as to improve new renewable and clean energy sources. To meet energy needs of the developed communities, the renewable energies can be used conjunction with energy storage systems. Also, efficient and compact energy storage systems seem to be important parameters to improve the use of renewable sources. [1].

Thermal energy could be stored as latent heat, sensible heat and thermo-chemical heat. Various thermal applications may include the thermal energy storage systems. For example; solar energy panels, heating and cooling systems and heat exchangers systems in industry contains thermal energy storage systems [2,3].

Latent heat storage materials which are known as Phase change materials (PCM) have a higher energy storage capacity with a small size of the systems. They absorb and release the heat at approximately constant temperature range during the phase transitions [4].

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To control the temperature in many engineering applications, phase change materials (PCM) are commonly used for thermal energy storage. According to their phase transition states, phase change materials are categorized in four groups. These categories are solid-solid, solid-gas, solid-liquid, liquid-gas PCMs [2, 5, 6]. Even though liquid-gas and solid-gas phase transition have higher latent heat capacity, the volume change during the phase transition is immoderate. This volume change makes the systems more complex and ineffective [2]. Because of this reason, solid-liquid and solid-solid phase changes are preferred and investigated from the many researchers. The solid liquid phase change materials are categorised in three main groups according to their chemical structure: inorganic, organic and eutectic. Although inorganic PCMs have more latent heat capacity than the organic PCMs, phase separation and subcooling may happen during the phase transition. The organic PCMs provide many advantages; for example, generally, they are not toxic, noncorrosive, and crystallize with no super cooling and segregation. Organic PCMs are examined in two groups: paraffin and non-paraffin [3- 6]. An eutectic mixture comprising two or more components forms as composition with a minimum melting point. During the crystallization every component in the mixture melts and freeze and the composition of the formed component crystals is compatible with each other [7]. The eutectic mixture may melt and freeze without phase separation because of their crystal structures. The components in the mixture melt at the same time without segregation [3].

Hydrate salts as inorganic PCMs, have some advantages of constant phase change temperature, higher heat storage capacity (nearly 300 J/g) comparatively high thermal conductivity (0.5 W/m°C) and moderate costs. They are also nonflammable and nontoxic. Thus, it may be more suitable and safer than organic PCMs in energy storage processes. The most researchers have studied with hydrate salts and eutectic mixture of the hydrate salts [8].

To prevent liquid leakage during the solid-liquid phase change, the porous materials are generally used as supports. Graphite, expanded graphite, aluminum powder, expanded perlite, activated carbon, and carbon nanotubes are chosen to keep off the liquid leakage.

Expanded perlite (EP) is an amorphous glassy volcanic rock and having porous structure. Because it has very low thermal conductivity (0.03 - 0.05W/m°C), low sound transition property and nonflammability it is generally used as insulation material in buildings (9, 10). Also, some researchers reported that, EP have a good supporting matrix for preparing composite PCMs. At the same time, PCM composites which are prepared by using expanded perlite exhibit a reduced thermal conductivity [10, 11, 12]. Li et al. have studied shape-stabilized PCMs by impregnating of paraffin into the expanded perlite pores. They used vacuum impregnation method and produced heat storage boards by adding gypsum into the prepared composite for use in buildings [13]. Z. Lu et al. prepared and examined form-stable PCMs and using the direct impregnation method to absorb the paraffin into the pores of the EP, their results showed that the leakage was prevented [14]. Karaipekli and Sarı studied Capric-myristic acid/EP composite as phase change materials they used vacuum impregnation method and the prepared composite PCM had good thermal properties and stable for thermal applications [15].

Although PCMs prepared with organic-organic eutectics and inorganic salts and their eutectic mixtures have been widely studied in the literature, there are not many studies about the organic-inorganic eutectics. The aqueous salt solutions are used as PCM for low temperature applications, but these may cause the erosion. In this research, an organic-inorganic eutectic mixture of Glycerol-Ni (NO₃)₂·6H₂O were studied for low temperature applications and form stable Glycerol-Ni (NO₃)₂·6H₂O /Expanded perlite composites were prepared by using wet impregnation and vacuum impregnation methods. Both methods are inexpensive and eco-friendly method for the composite PCM preparation. The

prepared Glycerol-Ni (NO₃)₂6H₂O eutectic mixture was enclosed into the porous of the EP and in this way the leakage was prevented during the phase transitions. Differential scanning calorimeter (DSC) and thermo gravimetric analysis instrument (TGA) were used to analyze thermal properties and stability of prepared composite. The surface area and pore volume of the EP and composite were determined nitrogen adsorption analyses for Brunauer-Emmett-Teller (BET). The structures and morphologies of the perlite and Glycerol-Ni (NO₃)₂6H₂O composites were determined by Fourier transformation infrared spectroscope (FTIR) and scanning electronic microscope (SEM). Heating and cooling cycle tests were performed to define heat accumulation and heat release properties of PCMs.

2. Experimental Study

2.1 Materials

Glycerol and the metal salt of Nickel (Ni(NO₃)₂6H₂O) were purchased from Merck. Expanded Perlite (EP) was supplied by the Batiçim-AS Company, Turkey. It was heated in an oven at 110°C before using.

2.2 Methods

Before beginning the composite PCM preparation, the eutectic point of mixture of glycerol and Ni(NO₃)₂6H₂O was calculated to decide the compositions which gives lower melting point. The Schroder equation (1) was used for calculation of the eutectic point [7].

$$\ln x_B = -\frac{\Delta H_B}{R} \left(\frac{1}{T} - \frac{1}{T_{mB}} \right) \quad (1)$$

where ΔH is latent heat, T_m is melting temperature as K, subscript B indicates the one of the components into mixture.

Then the PCM mixture was prepared in these compositions. The prepared PCM mixture included 97 wt. % glycerol and 3wt. % Ni(NO₃)₂6H₂O. Evaluated melting point of mixture at this concentration is -10 °C.

Initially, PCM mixture was prepared. Nickel salt was weighed and melted at 57°C in a backer and weighed glycerol was added in the salt and mixed during 1 hour, at 1000 rpm to obtain homogeny solution. To enclose the mixture into EP pore, two different methods were used (Wet impregnation and vacuum impregnation). Firstly, wet impregnation method was applied. EP was added to prepared mixture solution and the resulting slurry was put in an ultrasonic bath at 50°C for 30 minutes to penetrate the solution into the EP pores. Finally, the obtained composite was placed into an oven at 60°C for 10 hours. Secondly, vacuum impregnation method was used. Weighed EP was placed in the vacuum set and the prepared mixture was added it slowly under vacuum at 50°C. Then, the obtained mixture was placed into an oven at 60°C for 10 hours.

The experimental set up of the vacuum impregnation is shown in Figure 1.

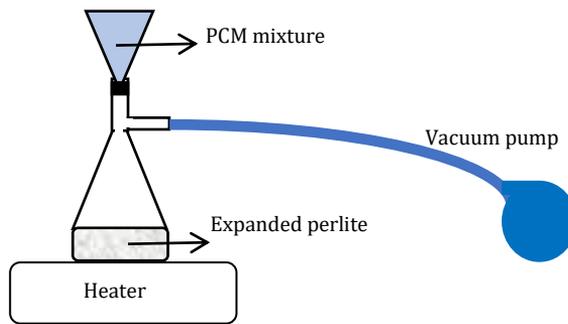


Figure 1 Experimental set-up for vacuum impregnation

To perform heating and cooling cycles, specific measure of prepared composite sample was placed into a glass tube and for recording the temperature a thermometer was put in the center of the tube. The tube was located in the hot water at 40°C for heating. When the temperature of the composite in the tube was reached steady state, the sample tube was carried into chilled water alcohol bath at -15°C for cooling. The temperature of the composite sample in the tube was measured with time while the heating and cooling process performed. 100 heating cooling cycles were performed to specify the prepared composite reliability.

3. Results and Discussions

3.1 Leakage Tests

To determine the absorption capacity of the EP for the eutectic mixture, leakage test was performed. Before the leakage test, PCM composite, containing 50 wt.% of perlite was prepared. This prepared PCM composite was placed on the filter paper and allowed to stand in an oven at 20°C for 30 min. The prepared PCM composite exhibits no leakage as shown in Figure 2.

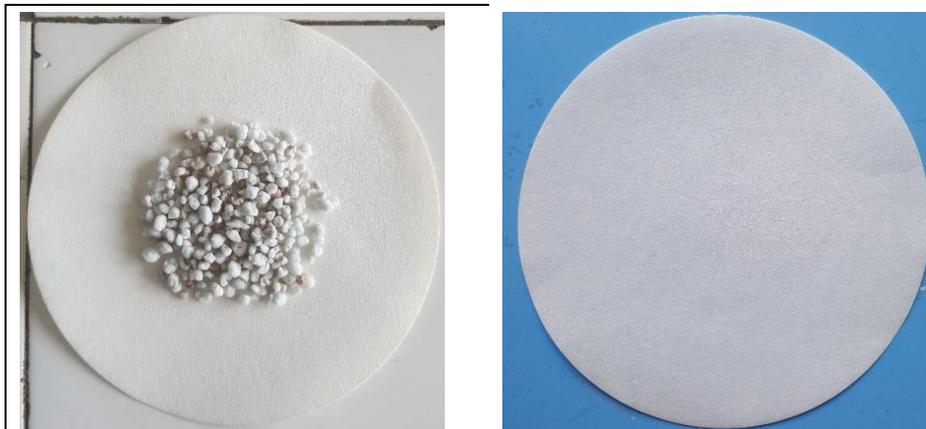


Figure 2. Images of the leakage test of the composite PCM

3.2 Characterization of chemical compatibility between the eutectic mixture and EP

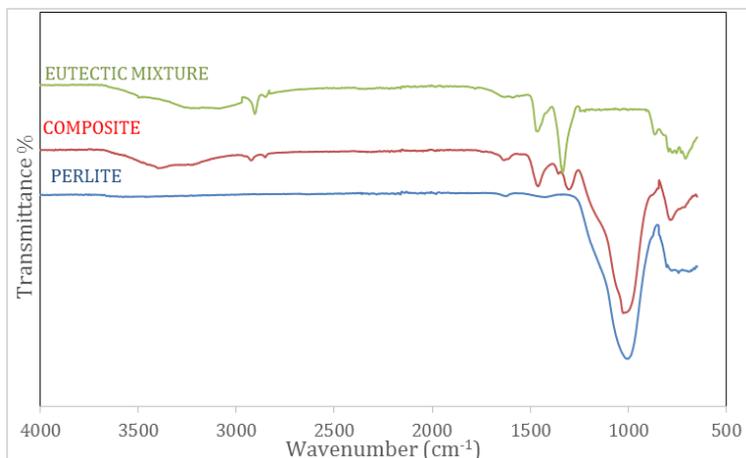


Figure 3. FTIR Spectrum of perlite, eutectic mixture and prepared composite PCM

The FTIR spectrum of expanded perlite, eutectic mixture and prepared PCM composite are shown in Figure 3. In the FTIR spectrum of the EP, the absorption band nearly at 850-900 cm^{-1} represents the vibrations of the Al-OH-Al and Al-OH-Mg groups, respectively. The intense and deep peak at 1020 cm^{-1} is described the Si-O-Si symmetric vibrations. The weak peak at 1625 cm^{-1} is described to the bending modes of the OH groups which are appended to the surface of expanded perlite and the adsorbed water molecules on the perlite [22]. In the FTIR spectra of PCM composite, which included perlite, glycerol and nickel salt, a medium absorption bands around 1300 cm^{-1} and 1360 cm^{-1} confirm the C-O stretching modes of glycerol. The symmetrical stretching vibration of CH₂ group which is stretching modes of glycerol is also shown at 2845 cm^{-1} . Intense and broad band in the range between 3000 and 3600 cm^{-1} shows the vibration of OH from coordinated those strong hydrogen bonds of water. The peak at 1458 cm^{-1} confirms the presence of the NO₃ from the nitrate salt in the composite. The band at 780 cm^{-1} can be assigned the Ni-O stretching vibration.

According to the FTIR spectrum of the composite, only the expanded perlite, glycerol and Ni(NO₃)₂·6H₂O peaks are seen, there are not observed new peaks. These specify that there is no chemical reaction between the prepared PCM mixture and the expanded perlite. The FTIR results show that the prepared composite is only a physical combination of the expanded perlite, glycerol and Ni (NO₃)₂·6H₂O.

3.3 Microstructure of expanded perlite and PCM composite

The BET surface area and micropore volume of EP and prepared composite were determined. According to the result the used expanded perlite BET surface area and pore volume are obtained 112.3 m^2/g and 0.63 cm^3/g respectively. After vacuum impregnation of eutectic mixture PCM into the EP pores, BET surface area and pore volumes were decrease to 0.0345 m^2/g and 0.000007 cm^3/g , respectively. These significantly decrease shows that the PCM mixture was placed into the EP pores.

Figure 4 a and Figure 4 b show the SEM images of the expanded perlite and composite PCM prepared by vacuum impregnation method, respectively. It can be seen from Figure 4-a,

the expanded perlite has a highly porous structure having many micro-pores with different sizes. These structures make the expanded perlite as a good supporting material to keep the PCM mixture. As seen from Figure 4-b, PCM mixture were dispersed uniformly in the porous and the sheets of the EP and the composite shows the most favorable homogenous morphology.

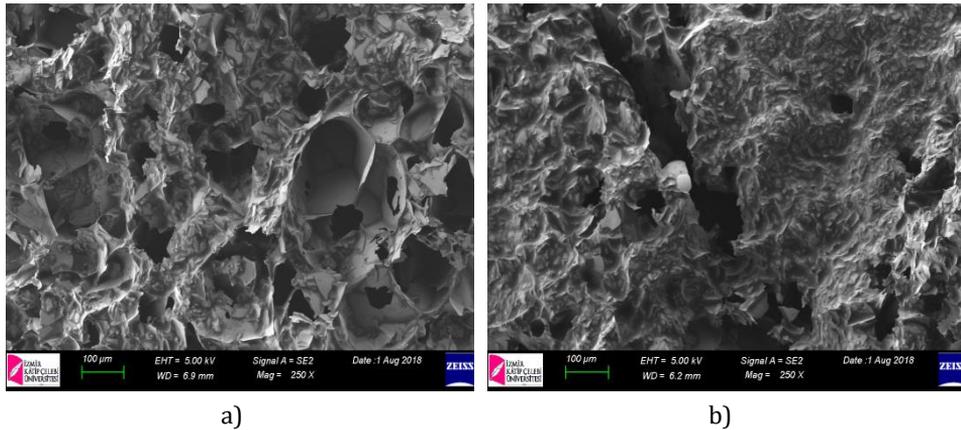


Figure 4. SEM images of a) expanded perlite, b) PCM composite prepared by vacuum impregnation method.

3.4 Thermal properties of eutectic mixture and PCM composite

The melting and freezing temperatures and latent heat values of the prepared PCM mixture and composites are listed in Table 1. DSC curves of PCM mixture and prepared composite with vacuum impregnation method before and after thermal cycling test are given in Figure 5. According to the DSC analysis from the PCM prepared using two different methods, there were observed no peaks in the wet impregnation method. It is thought that the PCM mixture could not be enclosed into the EP pores by using this method.

Table 1. Thermal properties of PCM

	Melting Point (°C)	Melting Latent Heat (J/g)	Freezing Point (°C)	Freezing Latent Heat (J/g)
PCM Mixture	-9.7	165	-9.8	163
Wet Impregnation	-	-	-	-
Vacuum Impregnation	-8.3	134	-8.2	136
Vacuum Impregnation (After 100 cycle test)	-8.4	130	-8.1	136

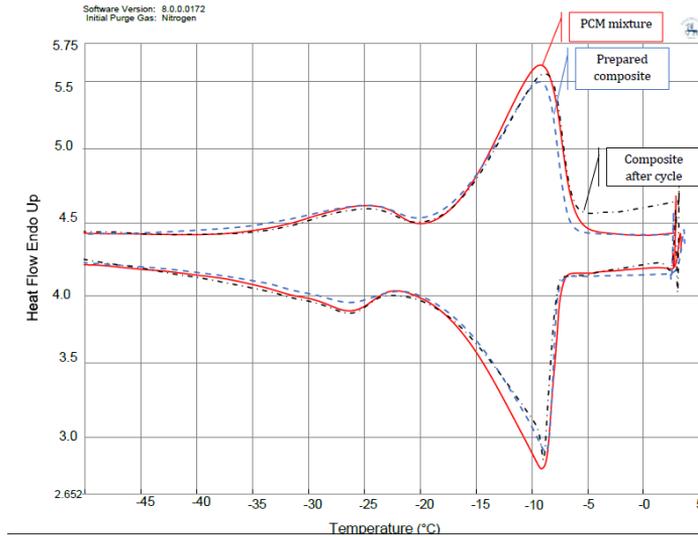


Figure 5. DSC curves for prepared composite PCM

According to DSC results, the phase transition temperatures of composite PCM which prepared by using vacuum impregnation method were about 1.4°C higher than that of the prepared eutectic mixture. On the other hand, the latent heats prepared composite PCM were found to be lower than the latent heats of the PCM eutectic mixture. The changes of the phase transition temperatures and the latent heat values are an indication of the force of the interactions between the expanded perlite and PCM mixture. When there is a strong relation between the PCM and pores of the support, the phase change temperature may increase and the same results have been found by some researchers for different composites [15, 16, 17 and 18]. The reduction of the latent heat value can be explained as the physical relation between the EP pores and eutectic mixture. C. Wang et al. have referred that the interaction between PCM and its support material cause the decrease of latent heat value [16]. Karaipekli and Sarı prepared eutectic mixture of capric and myristic acid and impregnated it into the EP by using vacuum impregnation method and they obtained form stable composite PCM. According to their results, melting and freezing temperatures of the eutectic mixture and prepared composite were determined as 22.61, 21.18 °C and 21.70, 20.70 °C, respectively [15]. Galazutdinova et.al. studied with inorganic composite mixture includes 40% of $MgCl_2 \cdot 6H_2O$ and 60% of $Mg(NO_3)_2 \cdot 6H_2O$. The prepared inorganic mixture, melting temperature and latent heat value were obtained as 62 °C and 120 J/g. Vacuum impregnation method was used to enclose the $MgCl_2 \cdot 6H_2O$ - $MgCl_2 \cdot 6H_2O$ mixture into EP. The prepared composite PCM had melting temperature of 63.5 °C and latent heat of 106.8 J/g [19]. Zang et all investigated $MgCl_2 \cdot 6H_2O$ - $CaCl_2 \cdot 6H_2O$ Eutectic/EP composite PCM. The prepared eutectic mixture included 15% $MgCl_2 \cdot 6H_2O$. The melting temperature and latent heat were determined as 23.9°C, 151.9 J/g, respectively. Composite PCM including 50 wt% of EP was prepared by using this eutectic mixture. The $MgCl_2 \cdot 6H_2O$ - $CaCl_2 \cdot 6H_2O$ /EP composite had exhibited a melting point of 23.5 °C and latent heat of 73.55 J/g, and there was no leakage seen after leakage tests [20]. Yilmaz et al. developed binary mixtures of tridecane and dodecane to be used as PCMs for thermal energy storage in

cooling applications. The melting point and heat of fusion of their PCM was found as -9.1°C and 145 J/g [21].

The thermal reliability is the important point to evaluate the PCM composite performance. The thermal properties of the materials can change after many thermal cycles and latent heat and phase change temperatures may change significantly. A suitable composite PCM should have steady thermal properties with many phase changes cycles without degradation [8, 10, 15]. DSC results show that, the latent heat value and phase change temperatures of the prepared composite PCM remained nearly constant after 100 heating and cooling cycles.

3.5 Thermal stability of PCM composite

TGA is commonly used to determine the thermal stability of the composites, In Figure 6, the TGA curves composite PCM is shown. The weight loss of the composite starts nearly 80°C . Due to the evaporation of glycerol the decomposition amount increased rapidly after 150°C this temperature and 58 % weight loss were measured at 350°C . Before 150°C , only 5 % decomposition is seen.

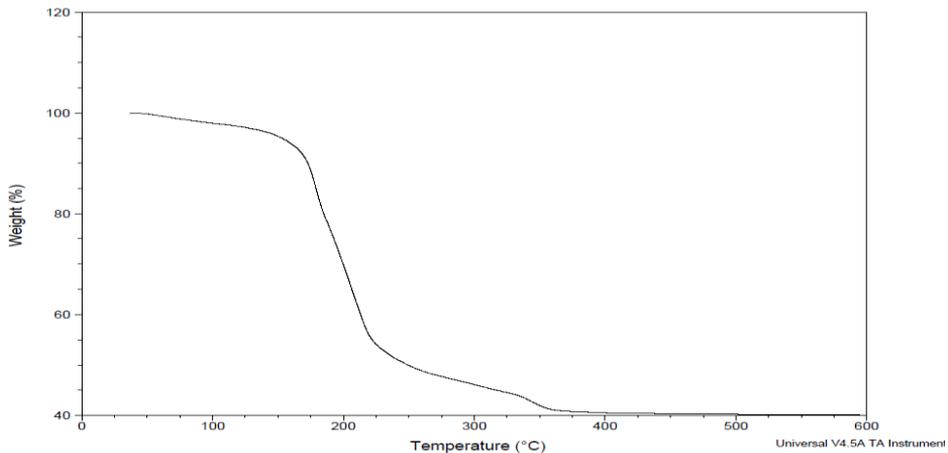


Figure 6. TGA curves of PCM composite

Heating and cooling cycles were performed to the PCM mixture and prepared PCM composite. The recorded results were drawn as temperature versus time graphic that is given in Figure 7. Each cycle of the heating and cooling were performed 20 min to obtain the steady state temperature. Figure 7 shows that, the heating and cooling rate of the prepared composite are lower than that of the PCM mixture.

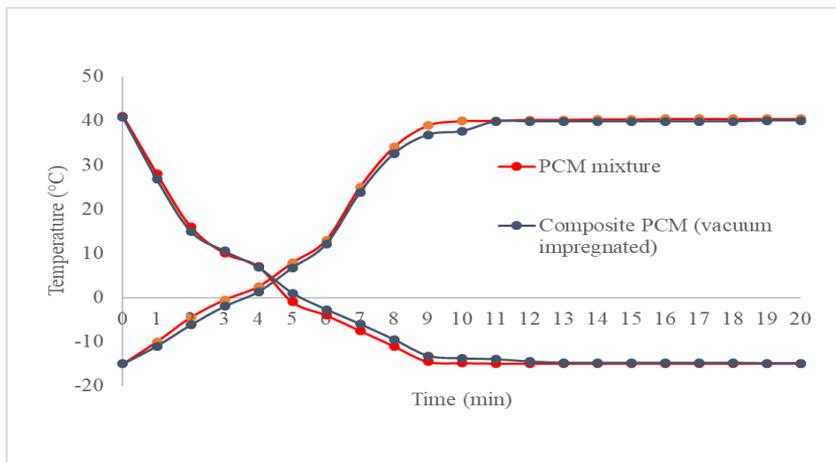


Figure 7. Heating and cooling processes for PCM mixture and PCM composites

4. Conclusion

In this work, Glycerol-Ni(NO₃)₂6H₂O / EP composite PCMs were prepared by using wet impregnation and vacuum impregnation methods. The used both methods are environmentally safe and economical therefore they are suitable for fabrication of PCM composite for industrial applications. But, according to the DSC analysis, there were not observed any phase change point by using wet impregnation method. It is thought that the PCM mixture could not be enclosed into the EP pores by using this method. Before preparation of the PCM mixture, eutectic point of the Glycerol-Ni(NO₃)₂6H₂O mixture was determined as melting point at -10°C, with 97 wt.% glycerol and 3 wt.% Ni(NO₃)₂6H₂O concentrations and the mixture were prepared at calculated composition, then it was absorbed into perlite pores.

The melting point of PCM mixture and PCM composite which prepared by using vacuum impregnation were found -9.7 and -8.3°C respectively. And phase change enthalpy values were determined 165 J/g for PCM mixture and 134 J/g for PCM composite. The changes of the melting and freezing temperatures are an indication of the force of the interactions between the EP and PCM mixture. The decrease on the latent heat values depending on the relation between the EP and PCM mixture. After heating and cooling cycles performed the composite PCM retained its melting and freezing temperatures and latent heat values. SEM images show that PCM mixture was distributed uniformly in the porous and the sheets of the EP and the composite shows the most favorable homogenous morphology. The leakage test show that the phase change process takes place in the pores of the EP. There were no leakages obtained. According to the FTIR results the eutectic PCM mixture and EP keep their original structure in the composite and the composite PCM was only the physical combination of nickel salt, glycerin and EP. After the heating cooling cycle, there was no leakage and degeneration at the composite PCM. TGA results revealed that the prepared composite has good energy storage capacities, good thermal reliability and stability.

Based on the obtained results, the presented composite PCM provide many properties of shape stabilization, moderate latent heat value and good thermal stability. In conclusion, it can be said that the prepared composite PCM has a great potential for the use in low temperature applications of thermal energy storage.

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