

Stearic Acid as Phase Change Material: Thermal Reliability Test and Compatibility with some Construction Materials.

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Abstract

An experimental study has been carried out to determine the thermal reliability of stearic acid (SA) as phase change materials (PCM). Thermal reliability of SA was analyzed as gradually change in melting temperature and latent heat of fusion of SA. 450 thermal cycles were carried out on SA in constant temperature hot air oven and after regular interval of thermal cycles melting temperature and latent heat of fusion was obtained by Differential Scanning Calorimeter (DSC). The DSC analysis shown that, there was no noticeable degradation in the thermal properties of SA. Thus the SA could be a promising PCM for latent heat storage systems. Some of the PCM has corrosive nature with some construction materials, so that compatibility of SA with construction materials (Stainless steel (SS304) and Brass) was analyze. The corrosive effect of SA was analyzed as the gravimetric analysis and metallographic analysis on construction materials. From the corrosive test it was observed that, SA has sewer corrosive effect on brass and insignificant corrosive effect on SS304 material.

1. Introduction

Solar energy is the huge reservoir of the thermal energy and it can be store as sensible heat and latent heat. Now a day, latent heat storage is the realistic option for thermal energy storage because it has large heat storage capacity compare to sensible heat storage. Latent heat can be store with phase change materials (PCM), which store heat during melting process and release heat during solidification process. For latent heat storage system the application of PCM requires a proper selection of PCM, because large numbers of PCMs are available in market with different thermal behaviour. Various organic, inorganic, salt hydrates and their eutectic mixture are studied by researcher from several decades [1, 2].

Recently several researchers are interested in fatty acid used as PCM because of its distinctive characteristic accepted some of the demerits as low

thermal conductivity and high volume changes during phase change [3]. Fatty acid has shown a narrow temperature range of melting and freezing phase transition. Fatty acid has the lowest corrosive effect on construction materials compared to salt hydrates due to that it is provide flexibility in application with different construction materials as PCM. It is also easily available in the commercial market since it is readily available in plastic, textile, and lubricant industries and many of them are formed from the renewable sources and oil seeds [4, 5].

Before application of PCM, it is essential to be familiar with the thermal properties and valuable life span of PCM for the latent heat storage system. As an ideal PCM it is desire that, there are no changes in melting temperature and latent heat of fusion with repeated melting-freezing thermal cycles. In a solar latent heat storage system only one melting-freezing cycle per day, this can be called as normal cycle. However to test the thermal reliability in terms of change in melting temperature and latent hear of fusion, a melting-freezing thermal cycles are conducted in laboratory called as accelerated thermal cycle. [6, 7]. Shukla A. et al. [8] has conducted thermal cycling test to check the thermal reliability of paraffin wax, sodium hydroxide, di-sodium borate, ferric nitrate, barium hydroxide and erythritol. 1000 thermal cycles were conducted on PCM and they were checked with Differential Scanning Calorimeter (DSC) for the variation in melting temperature and latent heat change. Sharma S. D. et al. [6] studied the change in melting temperature and latent heat of fusion of commercial grade stearic acid, acetamide and paraffin wax upto the 300 thermal cycles. Sari A. [9] studied the thermal reliability of stearic acid, palmitic acid, myristic acid and lauric acid as PCM with respect to 1200 thermal cycles. All of the PCM were with industrial grade 90-97%.

Some of the PCM has corrosive nature on some construction materials. So before the application of PCM it is essential to study about the corrosive effect of PCM on construction materials of the system. There is a very partial study on the

corrosion effect of PCM. Sari A. and Kaygusuz K. [10] has worked on some fatty acid like stearic, palmitic, myristic and lauric acid with industrial grade 90-95% purity. The thermal cycle test was conducted on PCM upto 910 thermal cycle and also studied the compatibility of PCM with construction materials like SS304 L, Steel C 20, Al and Cu. The compatibility of PCM was tested as gravimetric analysis (mass loss and corrosion rate) and metallographic analysis. Farrel J. et al. [11] has studied on corrosion effect of salt hydrates on metal piping, plate and housing of heat exchanger. The corrosion rate and metallographic analysis was examined on aluminium alloy and copper parts of heat exchanger and also discussed the way of preventing the corrosion. Garcia-Romero A. et al. [12] evaluated the corrosion behavior of glaubers salt ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) on four types of aluminium alloy.

The present study is deals with mainly two objectives. First objective is to carry out the 450 thermal cycle of Stearic Acid (SA) with industrial grade 95-99% purity. The analysis on the thermal properties of SA was considered as variation in melting temperature and latent heat of fusion. After every 50 thermal cycles melting temperature and latent heat was measured by Differential Scanning Calorimeter (DSC) for analyze the thermal reliability of SA. Second objective is to analysis the corrosion effect of SA on construction materials. The corrosion of construction materials was examined as gravimetric analysis and metallographic analysis upto 200 thermal cycles.

2. Experimental procedure

An experimental procedure has been carried out in two parts. In the first part the accelerated thermal cycling test is carried out on SA to examine the thermal reliability as change in melting temperature and latent heat of fusion with respected thermal cycles. In the second part the corrosion effect of SA was examined on stainless steel (SS304) and brass material as gravimetric analysis and metallographic analysis.

2.1. Thermal Cycling Test

The accelerated thermal cycles were carried out on SA. SA ($\text{C}_{17}\text{H}_{35}\text{COOH}$) is a fatty acid which was gained from S.D. Fine Chemicals Ltd., India, with 95-99% industrial grade. The quoted melting temperature of SA was in the range of 53-59°C. But actual melting temperature and latent heat of fusion of SA was 64°C and 159.71J/kg, which was examined by DSC.

2.1.1. Experimental setup. To carry out the accelerated thermal cycles a portable laboratory type constant temperature hot air oven was

designed and fabricated by Sensewell Pvt. Ltd, Vadodara, Gujarat. A digital thermostat is used to control the temperature range from ambient to 200°C with a resolution of $\pm 1^\circ\text{C}$. To measure the temperature precisely inside the oven, two K type thermocouples are used. The control panel indicate the inside temperature of oven. A relay switch is used to automatically control the constant temperature inside the oven. A glass window is provided on the opening door to observe the physical state of PCM. 200g of SA is packed in cylindrical bowl container, which made of stainless steel with internal diameter 9cm and height 4cm. This container is placed inside oven to conduct the thermal cycles.

2.1.2. Differential scanning calorimeter (DSC).

A DSC is used to obtain melting temperature and latent heat of fusion of SA. The DSC module manufactured by PerkinElmer Inc., USA is Jade DSC which is Perkin-Elmer's heat flux design. The Jade DSC is controlled by PyrisTM software.

2.2. Corrosion test

The corrosion effect of SA on construction materials as stainless steel (SS304) and brass plate (dimension 8.5×3.2×0.5cm) was examined upto 200 thermal cycles. Before the test SS304 plate was cleaned by 150 grain abrasive paper with thinner and brass plate was cleaned by 150 grain abrasive paper with water. Two cylindrical bowl containers (9cm internal diameter and 4cm height) were packed with SA and it was placed in oven for melting of SA. After complete melting of SA both plates were immersed individually in containers. These packed bowls were placed in oven individually to conduct the thermal cycle.

3. Analysis methods

To conduct the accelerated thermal cycle, the inside temperature of the oven was maintained at approximately 75°C for melting of SA. The melting was concluded when entire sample turned to liquid state. The solidification was started immediately after taking out the SA sample from the oven and allowed to cool at room temperature. 450 thermal cycles were conducted of SA and about 2g SA sample is withdrawn after every 50th thermal cycles for its DSC test. DSC graph indicate the melting temperature and latent heat of fusion after respective thermal cycle.

The corrosion test was conducted on the base of gravimetric analysis and metallographic analysis. After completion of every 50th thermal cycle, the SS304 and brass sample plates were taken out from the bowl and softly cleaned. The brass plate was cleaned with 100 grain abrasive paper by using water and SS304 was cleaned with same abrasive

paper by using thinner. The gravimetric analysis was performed as mass loss (mg/cm^2) and corrosion rate ($\text{mg}/\text{cm}^2\cdot\text{day}$) (If the SA is supposed to have only one thermal cycle in a day). After every 50 thermal cycles the weight of sample plates was measured with an accuracy of $\pm 0.01\text{mg}$. A metallographic analysis was carried out on the sample plates after 200 thermal cycles to analyze corrosion effect of SA. The Hitachi-Scanning Electron Microscope (S-400N) was used to analyze the corrosion effect of SA on Sample plates.

4. Result and discussion

4.1. Thermal reliability

Figure 4.1. show the DSC curves plotted by the DSC after 0^{th} , 50^{th} , 100^{th} , 150^{th} , 200^{th} , 250^{th} , 300^{th} , 350^{th} , 400^{th} and 450^{th} thermal cycles of SA. In DSC curve, the peak point of curve indicates the melting temperature and area under curve indicate the latent heat of fusion. The value of melting temperature and latent heat of fusion at 0^{th} thermal cycle was considered as reference. Table 4.1. show the measured melting temperature and latent heat of fusion of SA after 0, 50, 100, 150, 200, 250, 300, 350, 400 and 450 thermal cycles.

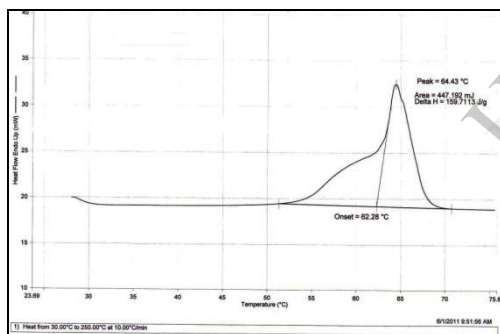


Figure 4.1. DSC curve of SA at 0^{th} thermal cycle

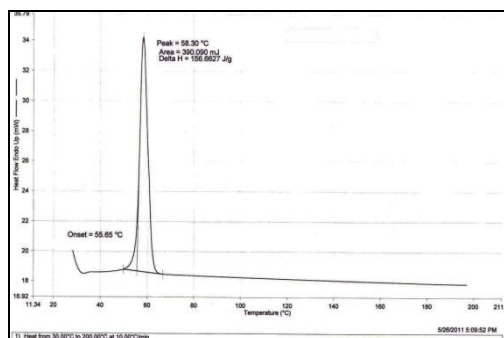


Figure 4.2. DSC curve of SA at 50^{th} thermal cycle

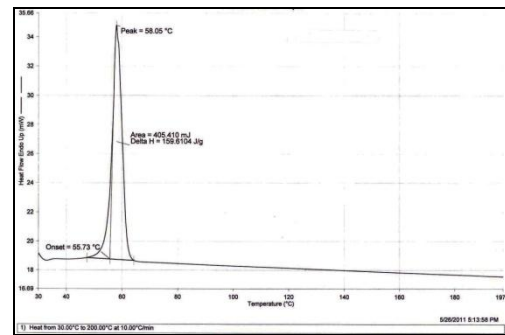


Figure 4.3. DSC curve of SA at 100^{th} thermal cycle

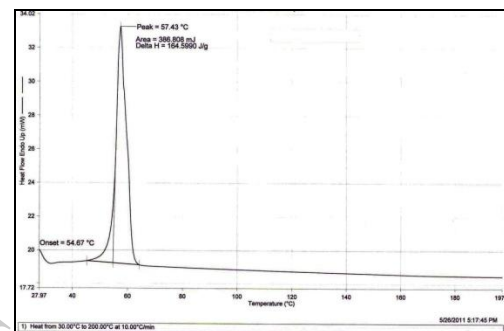


Figure 4.4. DSC curve of SA at 150^{th} thermal cycle

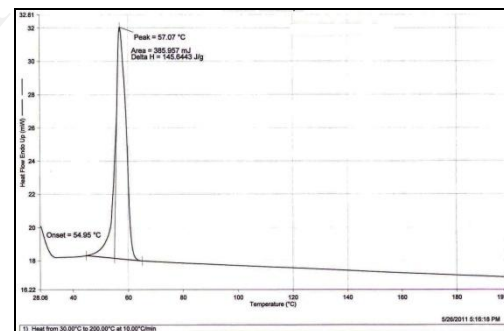


Figure 4.5. DSC curve of SA at 200^{th} thermal cycle

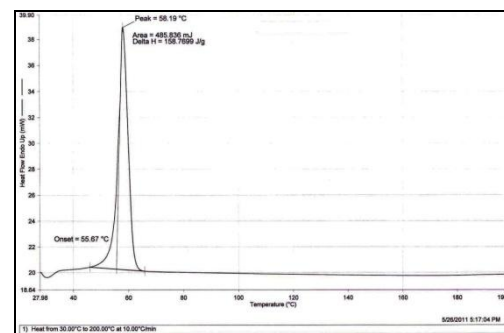


Figure 4.6. DSC curve of SA at 250^{th} thermal cycle

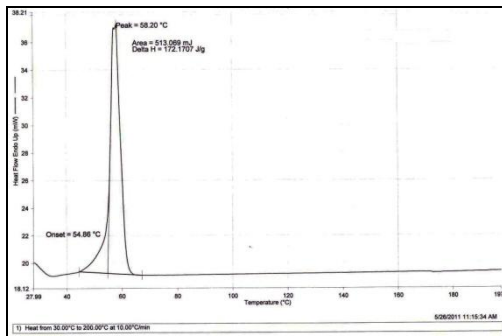


Figure 4.7. DSC curve of SA at 300th thermal cycle

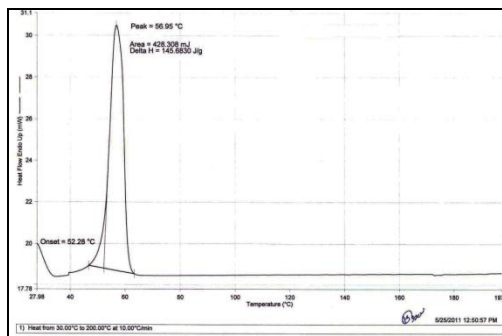


Figure 4.8. DSC curve of SA at 350th thermal cycle

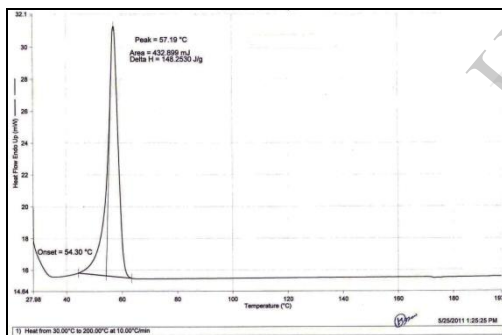


Figure 4.9. DSC curve of SA at 400th thermal cycle

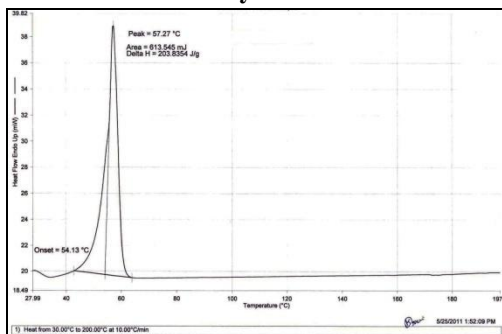


Figure 4.10. DSC curve of SA at 450th thermal cycle

Table 4.1. Melting temperature and latent heat of fusion at various thermal cycles

No. of Thermal Cycles	Melting temperature (°C)	Latent heat of fusion (kJ/kg)
0	64.43	159.71
50	58.30	156.66
100	58.05	159.61
150	57.43	164.59
200	57.07	145.64
250	58.19	158.76
300	58.20	172.17
350	56.95	145.68
400	57.19	148.25
450	57.27	203.83

Table 4.1. show that the latent heat of fusion of SA at different number of thermal cycle, which was irregularly varies with respect to 0th thermal cycle of SA. This irregular variation in latent heat of fusion was because of its impurities or by using a very little sample of SA for DSC test. The melting temperature of SA was varying 9.51 %, 9.66% and 11.11% at 50th, 300th and 450th thermal cycles respectively as compared to 0th thermal cycle of SA. Variation in melting temperature and latent heat of fusion of SA with number of thermal cycles were irregular as well as invisible which shows reasonable good thermal reliability of SA as PCM. If 300 thermal cycles are conducted in a year then approximately 1.5 year utility period is an accepted level for the application of SA as PCM in a latent heat storage system.

4.2. Compatibility with construction materials

Figure 4.2.1. show the brass plate immersed in SA before the thermal cycles and figure 4.2.2. show the brass plate immersed in SA after 200 thermal cycles. It was found that the colour change of SA due to dezincification of the brass plate. There was no significant reaction between the SA and SS304 plate.



Figure 4.2.1. Brass plate immersed in SA before thermal cycle



Figure 4.2.2. Brass plate immersed in SA after 200 thermal cycles

For the corrosion test of SA, the result obtained from the gravimetric analysis as a mass loss (mg/cm^2) and corrosion rate ($\text{mg}/\text{cm}^2.\text{day}$) are summarized in table 4.2.1.

As observed from table 4.2.1, the mass loss for SS304 plate is found insignificant compare to brass plate. It was because of some impurities only in the surface of SS304, after 100 thermal cycles the corrosion rate of SS304 was observed zero. The highest corrosion rate was observed $3.92 \times 10^2 \text{ mg}/\text{cm}^2.\text{day}$ for brass plate at 50th thermal cycle but it was not regular during proposed thermal cycles. It was concluded that, because of the corrosion products and dezincification of the brass plate the mass loss was occurred.

4.2.1. Mass loss and Corrosion rate for plates

Sr. No.	Number of thermal cycles	Brass plate		SS304 plate	
		Mass loss (mg/cm^2)	Corrosion rate ($\text{mg}/\text{cm}^2.\text{day}$)	Mass loss (mg/cm^2)	Corrosion rate ($\text{mg}/\text{cm}^2.\text{day}$)
1	50	1.96	3.92×10^{-2}	0.45	0.9×10^{-2}
2	100	1.06	2.12×10^{-2}	0.15	0.3×10^{-2}
3	150	1.51	3.02×10^{-2}	0	0
4	200	1.96	3.92×10^{-2}	0	0

Figure 4.2.3. show SEM image of the brass metal sample before the thermal cycle and figure 4.2.4. shows the brass metal sample after 200 thermal cycles.

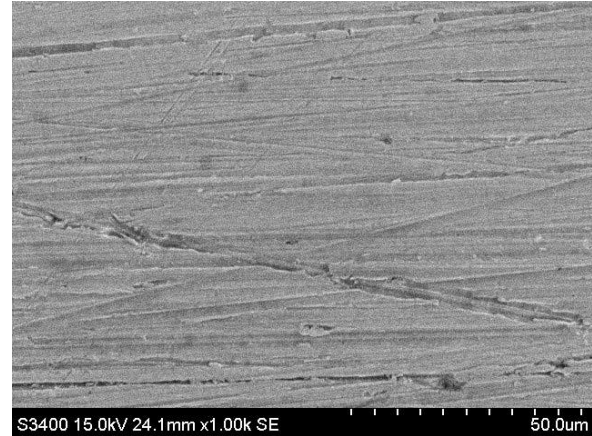


Figure 4.2.3. SEM image of Brass plate before thermal cycle

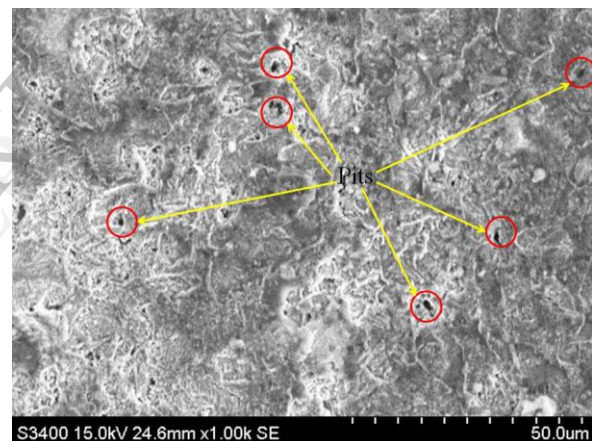


Figure 4.2.4. SEM image of Brass plate after 200 thermal cycles

Figure 4.2.4. shows the scratches on the surface of the brass plate, because of 150 grain abrasive brass plate was used to remove the unwanted layers

from the plate before starting the thermal cycle. It was observed from figure 4.2.4, after 200 thermal cycles the pits are observed on the surface of plate. The pits are irregular with respect to surface and they were not so deep. The corrosion products were also observed on the surface of the brass plate. There was negligible corrosion effect of SA on SS304 metal sample.

5. Conclusion

The experimental result shows that the change in melting temperature and latent heat of fusion was accepted for SA upto 450 thermal cycles. The deviation in melting temperature and latent heat of fusion of SA may be having impurities and improper crystallization during thermal cycle of SA or as only small amount of SA sample used for DSC test. Thus it can be concluded that SA have reasonable good thermal reliability as PCM used for latent heat storage system.

It could be also concluded that, before using SA as PCM it is essential to conduct the corrosion test of SA with construction materials of system. From the corrosion test the high corrosion rate was observed for the brass plate compared to SS304 plate. Because of the corrosion products and dezincification of the brass plate the mass loss was occurred. The SS304 had insignificant corrosion effect of SA.

6. References

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