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The Effect of Liquidus Aging on the Performance of Phase-Stabilized Wax with Solid Nano Additives

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Abstract: The present work assesses phase stabilized HW (hydrocarbon wax) with nano additives through liquidus aging treatment. The aging treatment is performed by storing the sample in the liquid phase at 130°C for 250 hours. The treatment is designed to imitate the actual process of thermal energy storage (TES) system which experiences continuous operation. It helps to evaluate the TES material for long-term operation consideration. The sample performance is assessed according to the heating and cooling rate change before and after aging treatment. The finding indicates a severe decrement in the heating rate of thermal conductivity enrichment (TCE)-HW up to 24.4% and 7.5% for the discharge rate. The phase-stabilized HW performs better according to its heating rate, which only decreases by around 10.9% and the discharge rate by only 1.2%. The heating profile for HW shows a distinctive phenomenon, indicated by a two-step temperature spike of 6.8°C and 11.8°C at the solid-solid and solid-liquid transition. Contrary to that, the SHW presents a suitable profile where the temperature increases steadily until 86.3°C with the average heating rate around 2.97°C/min. The surface observation shows that the phase-stabilized polyethylene (HDPE) decreases the potential of void formation which correspond to the plasticity effect. As a result, the SHW maintains suitably the distribution of nano additives after aging treatment. Thus, phase stabilization is critical to ensure stable operation of HW with nano additives for the TES system. Moreover, the finding can be used to mitigate the potential performance deterioration for TES system which uses thermal enrichment.

Keywords: dendrite, hydrocarbon wax, phase stabilizer, polyethylene, solid additives.

1. Introduction

The energy crisis is a global issue that forces us to develop an advanced technology system to explore and harvest alternative and renewable energy sources [1]. The effort has made a notable outcome that can produce various types of energy storage. The high-capacity electric battery for electric vehicles significantly reduces fossil fuel usage, making it a suitable method to decrease dependency in non-renewable sources. Another type of energy storage is thermal energy storage (TES), which plays a critical role in improving the thermal system's operation, especially for stationary applications [2]. It can be combined in processing heat and water heaters, which comes from alternative fuel [3]. Thus, the combined effect leads to a higher thermal efficiency [4].

TES is highly feasible in modern energy systems as it reduces the consumption of electrical energy. It utilizes the heat of fusion from storage material [5]. The stored heat is proportional to the latent heat and solid-liquid transition from the material [6]. As a result, the system can be operated at a minimum temperature gradient, which is advantageous to adjust the system's operation accordingly [7]. Moreover, the stored heat can be increased by employing the sensible properties of the storage material in the liquid state. It allows the system to store more energy per unit volume, promoting better volumetric efficiency from the storage system.

The organic storage material, such as hydrocarbon wax (HW), is the ideal candidate for TES material operating at moderate temperatures [8]. It is compatible with metal storage containers, economically feasible and widely available. HW is generally chosen as the phase change material (PCM) for TES application. There is one particular problem during the operation of HW in the TES system. Its poor conductivity makes the heat transfer rate extremely slow and reduces the overall performance of the TES system [9-11]. It is affected by the major constituent of HW, which is solely composed of organic material with a low thermal conductivity (TC). A specific approach is taken to encounter the issue by adjusting the storage unit [12], modifying the heat exchanger [13], and the liquid flow within the system [14]. The proposed method positively influences the system's operation, but it is only suitable for specific applications and requires extensive adjustment if applied in different storage systems.

Materials science has a significant impact to encounter the operational problem of HW in the TES system. It can be done by adding solid additives to accelerate the heat transfer rate for HW [15]. B. M. Suyitno et al. [16] employed volcanic ash as solid additives and indicate the increment in TC up to 19.6 W/m:K. The addition of smaller quantities of solid additives is focused on maintaining the storage capacity. One convenient method can be taken by using metallic filler. H. Hosseininaveh et al. added Fe_3O_4 as metallic particles with a 1–4 wt% ratio and increased the solidification rate for the PCM around 4%–20% [17]. Non-metallic solid particle such as carbon nanotubes (CNT) is also desirable to improve the heat transfer characteristic of PCM [18-20]. The CNT can be synthesized in a sustainable way, which ensures a better sustainability of the material. It motivates the researcher to utilize CNT as a solid additive for HW in the TES system.

The operation of HW in the TES system forces the material to experience solid–liquid phase change [21]. The cycle is repeated as the system operates, causing the aging effect that deteriorates the system's performance for an extended period [22]. The solid additive has a higher density than HW, which probably causes redistribution within the storage tank after a specific cycle. It can be seen from previous work which utilizes solid additives for the TES system. For example, modification of pressing direction for HW/expanded graphite is required to maintain the storage system's performance due to a substantial decrement in the thermal capacity of around 3% after 100 cycles [23]. To overcome the issue, metal matrices can be employed for the storage tank [24]. However, it reduces the net volume of the storage material, which causes side effects regarding the volumetric energy ratio [25].

Alternative methods can be taken to solve the degradation of the HW system with solid additives by introducing a supporting matrix. Several supporting matrices can be used, such as silicone-based [26], copolymer [27] and polyethylene (high density/HDPE) [28]. The supporting matrix stabilizes the operation of HW, especially at extended cycles. Compared to others, HDPE has the highest feasibility since it has a suitable viscosity and melting behavior [29], providing a better thermal response. Therefore, combining stabilizers as phase stabilization and solid additives as thermal conductivity enrichment (TCE) is expected to improve the operation of HW in the TES system.

One critical aspect for the TES system is long-term operation. Repeating the cycle between the solid and liquid phases eventually reduces the performance of TCE–PCM. Thus, further study is required to analyze the phenomenon and observe the role of stabilizers in PCM systems with solid enhancers. The change in performance, including the detailed profile temperature behavior is essential to mitigate the impact of the repeating cycle and its relation for the TCE-PCM. The outcome of this work can be used as a specific reference to adjust the operation of the TES system, especially for the extended process that requires a high operation cycle at elevated temperatures in the liquid phase of PCM. The suitable adjustment can be taken by understanding the performance behavior and helps to ensure the maximum performance of TES system, especially for the large-scale operation which is essential to maintain the peak performance of the thermal system.

2. Materials and Methods

The synthesis process of stabilized HW (SHW) is shown in Figure 1a. Commercial grade HW was heated up to 110°C to remove moisture content within the substance. The HDPE was melted separately at a higher temperature (170°C) to ensure an ease mixing process. The molten material was mixed at a ratio of 85:15 (weight-based). The mixture was then reheated until it melted again, and then 5 wt% of CNT (average diameter 10 nm) was added to the molten PCM. The sample was stirred adequately and cooled back to room temperature. The mass of the PCM was set at 20 grams.

The liquidus aging process was done by continuously maintaining the sample at the liquid phase. The sample was filled into the test tube and submerged in an oil bath (Figure 1b).

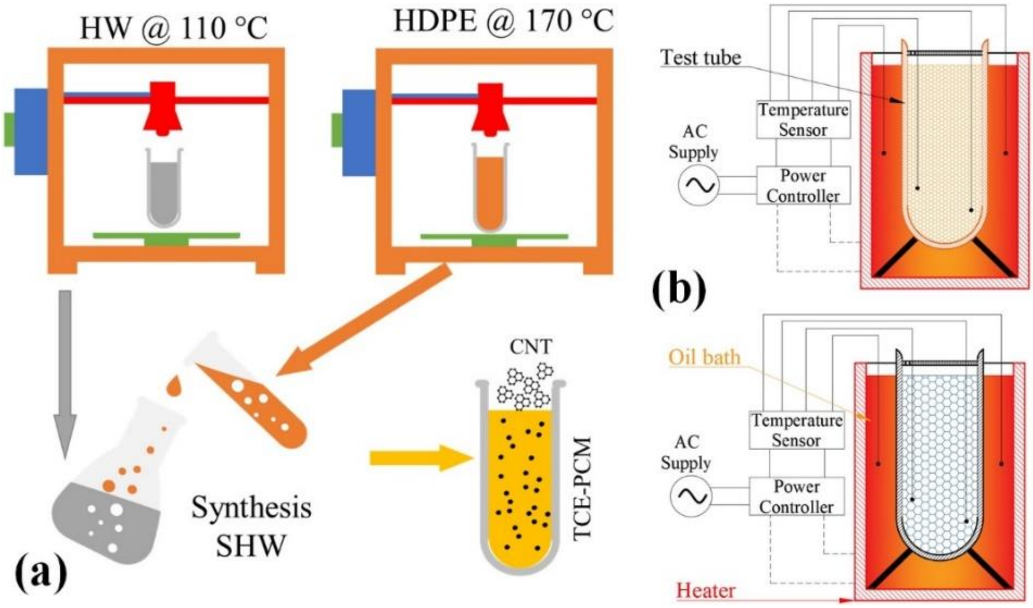

Figure 1. *a) Synthesis process of SHW and TCE-PCM, and b) Liquidus aging treatment of the samples*

The oil was heated by a heater and set at 130°C ± 5°C. The temperature of the oil bath and samples were monitored in four locations to ensure steady thermal diffusion between the heater, oil and sample. In addition, a copper tube was chosen as a test tube to maximize the heat distribution. Once the sample entirely melted, it was kept heated for 250 hours. After that, a static heating and cooling test was performed for the aged sample and compared before treatment. The temperature and time for each process can be used to estimate the average heating/cooling rate which is based on T-t method [30] and IEA-ECES Annex 30 recommendation [31]. Therefore, the temperature and time characteristics of the sample can be evaluated effectively.

The chemical composition of HW and SHW was characterized using infrared spectroscopy (FTIR). The phase and crystal profile for TCE-PCM were analyzed using a diffractometer (XRD). Also, the sample's morphology before and after the addition of solid additives was observed using SEM. Thermal behavior for the HW and SHW were investigated through scanning calorimetry and thermogravimetric methods. The material's properties were used for further analysis of the heating and cooling characteristics before and after liquidus aging treatment.

3. Results and Discussion

Phase stabilization of HW is performed to provide a better phase change during the operation, particularly for solid-liquid transition. The chemical composition of HW and SHW was observed to evaluate the effect of polymer-based stabilizers within the sample. As displayed in Figure 2, each material has an identic pattern according to the spectra profile. The first spectra peak indicates the stretching vibration of $–CH_2$ at wavenumber 2750–3000 cm^{-1}. The observed next peak appears in the deformation region $–CH_2$ and $–CH_3$. The latter peak demonstrates the rocking vibration of $–CH_2$. According to the result, HW is compatible with the polymer stabilizer. There is no new peak observed for the SHW. It can be concluded that the chemical characteristic of SHW remains the same.

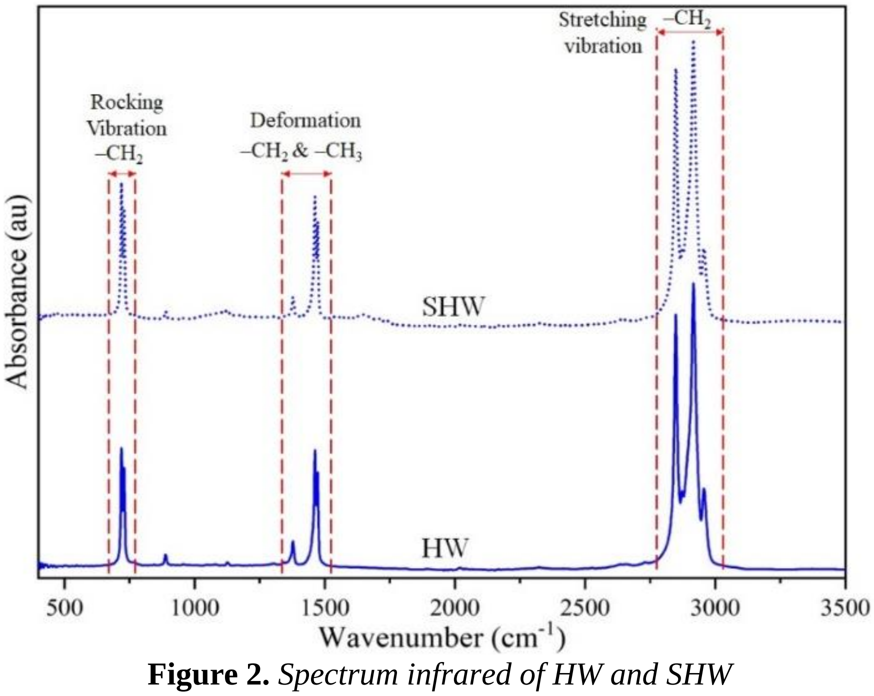

Figure 2. *Spectrum infrared of HW and SHW*

The solid additive (CNT) is a non-organic matter that requires a different approach to observe its role in the sample. Thus, the effect of TCE for HW and SHW is assessed through an X-ray diffractometer (XRD) to obtain the sample's crystal profile. Figure 3 indicates the main peak for the samples are observed at 2θ = 21° and 24°, confirming the typical profile of pristine HW and SHW [32]. The addition of a solid additive (CNT) for TCE does not alter its phase and crystal structure. Thus, the TCE is mixed physically within the sample without affecting the phase structure of the PCM.

Figure 4 presents the morphology of the PCM. HW shows a distinctive profile with the appearance of a large crater (Figure 4a). The crater signifies the poor solidification rate of hydrocarbon, resulting in void formation and a corrugated profile. It is affected by inhomogeneity nucleation growth [33] due to low intermolecular forces during the solidification. The role of stabilizer can be observed notably in Figure 4b. The plasticity effect of polyethylene protects the HW from void formation. It implies that the solidification rate of SHW is increased, which makes the positive outcome of the stabilizer provide a better phase transition.

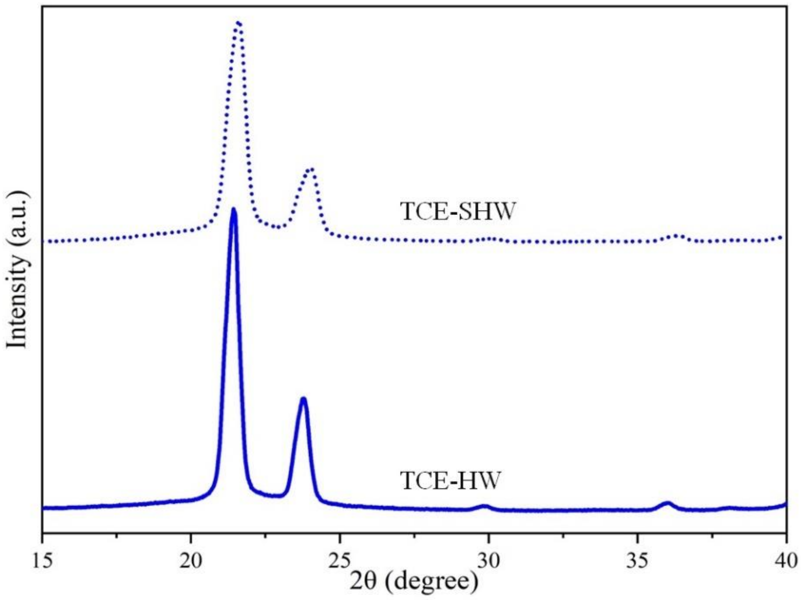

Figure 3. *Diffraction profile for TCE-HW and TCE-SHW*

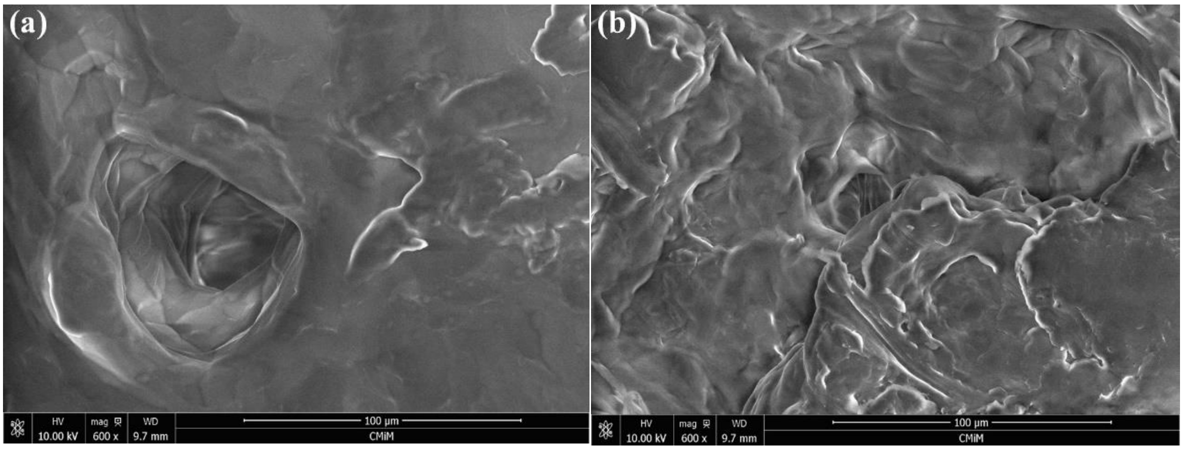

Figure 4. *Morphology of HW and SHW*

Further observation is taken for PCM with TCE. Figure 5a displays the unique profile of TCE–HW, which indicates the equiaxed dendrite appearance. The partial freezing process during solidification probably leads to this phenomenon [34]. Moreover, the formed dendrite is highly affected by the rapid heat transfer rate in the region due to the addition of solid additives. Therefore, adding TCE improves thermal diffusion within the HW structure. The stacked profile for TCE-SHW (Figure 5b) indicates the presence of local agglomeration, which can be affected by the polymer-based stabilizer. The liquid phase of the PCM influences the distribution of solid additive (TCE), which means local agglomeration potentially affects the SHW during the operation.

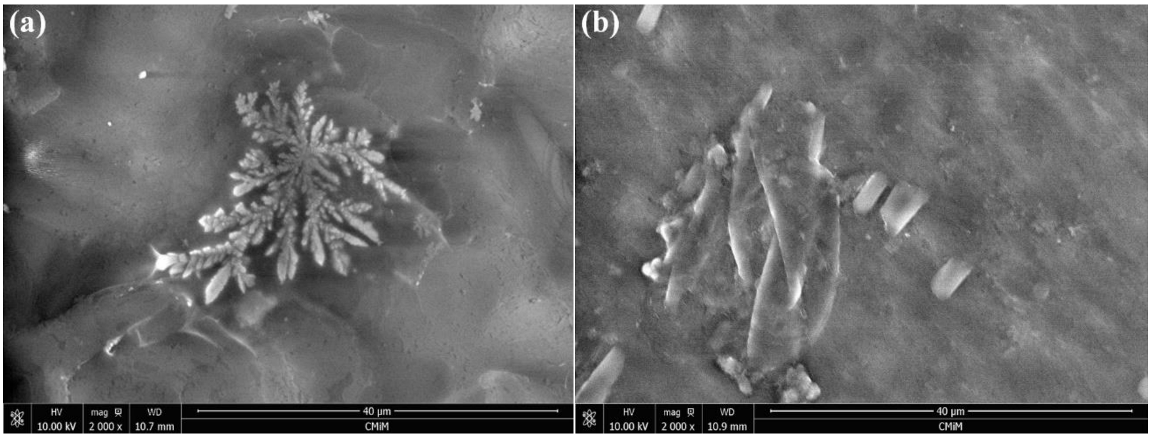

Figure 5. *Morphology of HW and SHW with solid additives TCE*

The solid-liquid transition of HW and SHW occurs at a closed temperature range between 59.16°C – 58.82°C (Figure 6a). The heat of fusion at the given region is obtained at 151.6 J/g (HW) and 149.1 J/g (SHW). The SHW has a lower heat of fusion due to the presence of a stabilizer. However, it has a new peak at a temperature 113.2°C with a melting enthalpy of 24.4 J/g. Therefore, the cumulative melting enthalpy of SHW is slightly higher than HW, making it more suitable to operate at elevated temperatures to reach the melting transition of the stabilizer. Moreover, the third peak indicates the phase segregation between HW and stabilizer, which causes the melting process to take consecutively.

The solidification involves intermolecular forces to release the heat. However, the poor nucleation growth of the hydrocarbon compound causes supercooling [35]. As seen in Figure 6b, the exothermic peak for HW is slightly shifted at a temperature 54.1°C. As a result, HW has a supercooling degree of 5.94°C. A similar condition is also observed for the latter peak of SHW, indicating the solidification takes at a temperature 103.1°C. However, the principal peak can be maintained at a suitable temperature of 54.8°C, implying the notable effect of phase stabilization for SHW. It means the supercooling degree can be set at a lower value for SHW. It proves the role of stabilizer, which promotes a better phase change, significantly reduces the probability of the partial solidification process of HW.

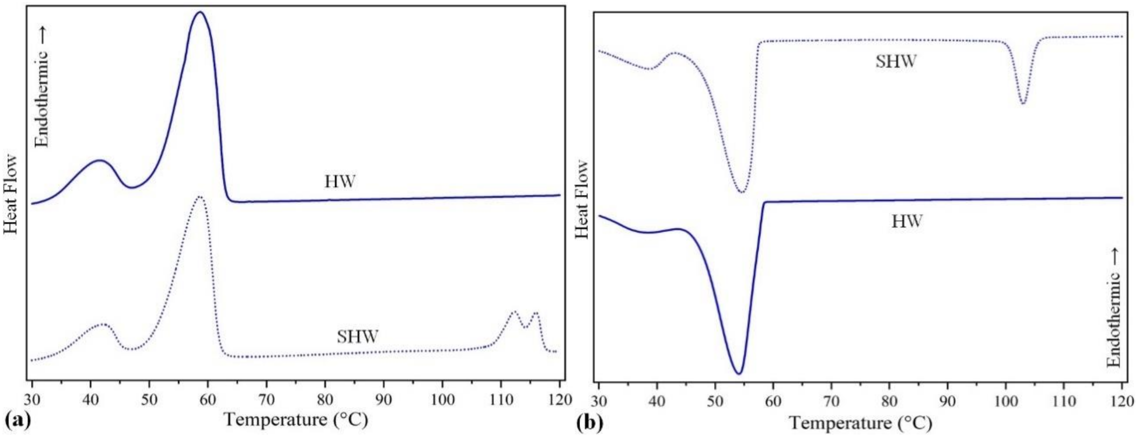

Figure 6. *Heating (a) and cooling (b) profiles of HW and SHW*

The decomposition profile for both PCMs is presented in Figure 7. The HW exhibits a direct decomposition, starting at 190°C and ending at 355°C. Interestingly, the decomposition process of SHW runs in two different steps. The first stage occurs between 200°C – 368°C, confirming the degradation of HW. The second stage is occurred between 420°C – 492°C. It demonstrates the decomposition process of the HDPE. The two-step degradation of SHW supports the DSC profile, which shows an additional peak at a temperature above 100°C. Thus, the addition of a stabilizer notably affects the thermal properties of HW.

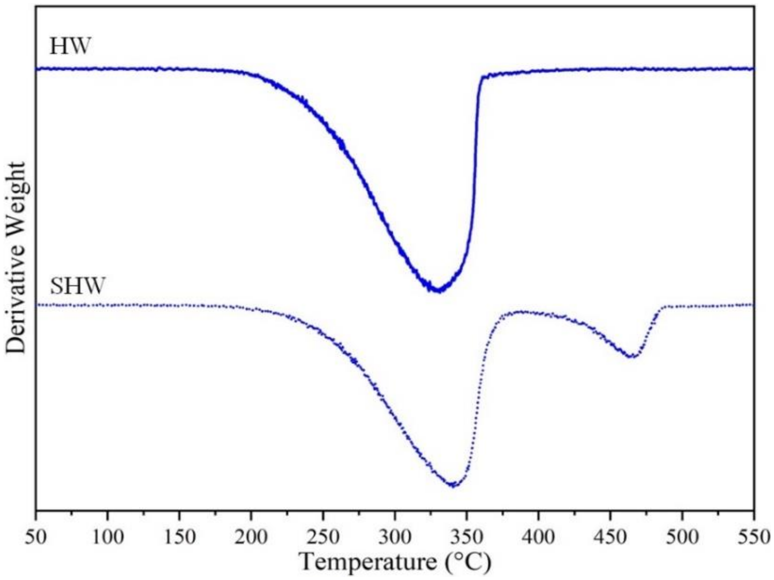

Figure 7. *The decomposition rate of HW and SHW*

The heating characteristic for both PCMs is shown in Figure 8. The temperature pattern for TCE-HW before aging indicates two contrast temperature spikes between 41.3°C – 48.1°C and 56.1°C – 67.9°C. The region is close to the solid–solid transition of HW according to the first peak of the DSC profile (Figure 6a), which occurs at temperature 41.7°C. The second spike is also related to the main endothermic peak of HW. Thus, both temperature spike is highly correlated with the phase transition of HW. The addition of TCE causes a rapid temperature increment of HW during the melting process. As a result, the temperature rises quickly during the heat uptake process of the HW. The high temperature spike is unfavorable which makes the charge process unstable. It is undesirable for most TES system, particularly for temperature-sensitive application such as thermal regulating system [36].

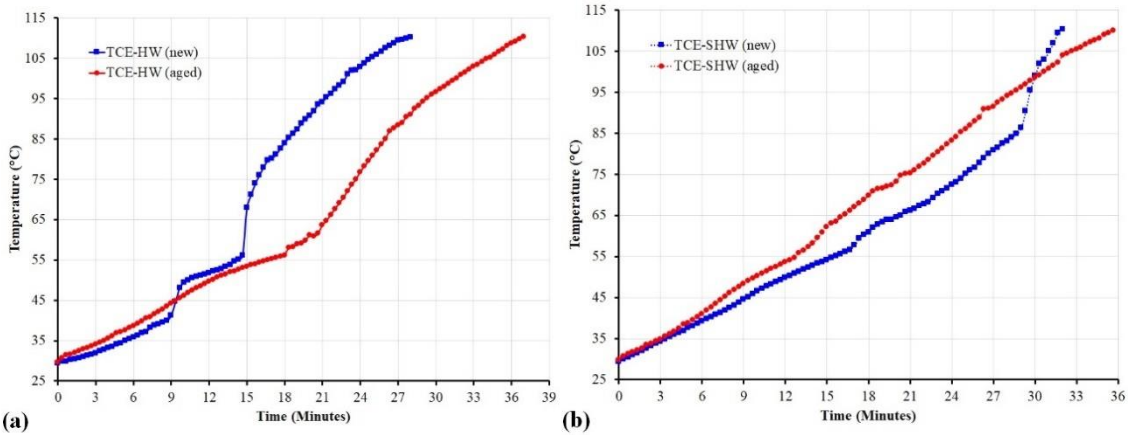

Figure 8. *Temperature increment before and after aging for HW (a) and SHW (b)*

The liquidus aging process is significantly affected the heating process of HW. There is no sudden temperature increment where the profile demonstrates a steady heating process. It implies that the distribution of solid additives changed due to liquidus aging, which causes a different heating profile of HW. The aging causes a slower heating rate of only 2.18°C/min, much lower than the average heating rate before aging (2.89°C/min). It explains the significant drawbacks of aging process that causes heat distribution within the PCM, particularly for the HW-based PCM [37].

The heating characteristic for TCE–SHW shows a steady profile without indicating a temperature spike. The temperature increases steadily for the TCE-SHW before aging process until 86.3°C. The polymer stabilizer promotes a better heat distribution, which minimizes the effect of phase transition and results in a stable heat increment profile. After that, the temperature is increased notably until reaching the maximum temperature range of 110.4°C. The region is close to the phase transition of HDPE (third peak in Figure 6a). Therefore, the effect of solid additives in this case can be taken as an acceleration of phase transition for HDPE.

The impact of liquidus aging is observed distinctively during the heat releasement stage. The cooling process for TCE-HW before and after liquidus aging is relatively identic, where the temperature falls immediately up to 57.15°C (Figure 9a). The average cooling rate for both samples is 4.8°C/min. The rapid heat release at the given temperature demonstrates a critical role of TCE, which helps to accelerate the discharge stage of HW during the liquid phase until fully solidified. However, the cooling rate decreases significantly once the HW reaches its solidus point.

The slow cooling rate typically occurs for solid HW. The heat is released for 54 minutes with an average cooling rate of only 0.39°C/min. Also, the cooling rate gets slower for the TCE-HW after liquidus aging treatment. The aged TCE-HW has a slightly lower cooling rate with only 0.36°C/min. The decrement in the cooling rate for aged TCE-HW can be affected by the change in the dispersion of solid additives. As a result, the crystallization is initiated in a localized region, reducing the average cooling rate. Thus, the positive outcome for TCE cannot be maintained effectively to improve the cooling rate for HW after liquidus aging treatment.

Phase stabilization for HW promotes a better discharge process. As presented in Figure 9b, the initial cooling profile is relatively similar to HW, indicating a rapid temperature decrease up to 57.8°C. The role of phase stabilization is observed clearly after passing the solidus point. The average cooling rate for TCE–SHW before aging is 0.41°C/min and can be improved after aging process, resulting in a higher cooling rate of up to 4.22°C/min.

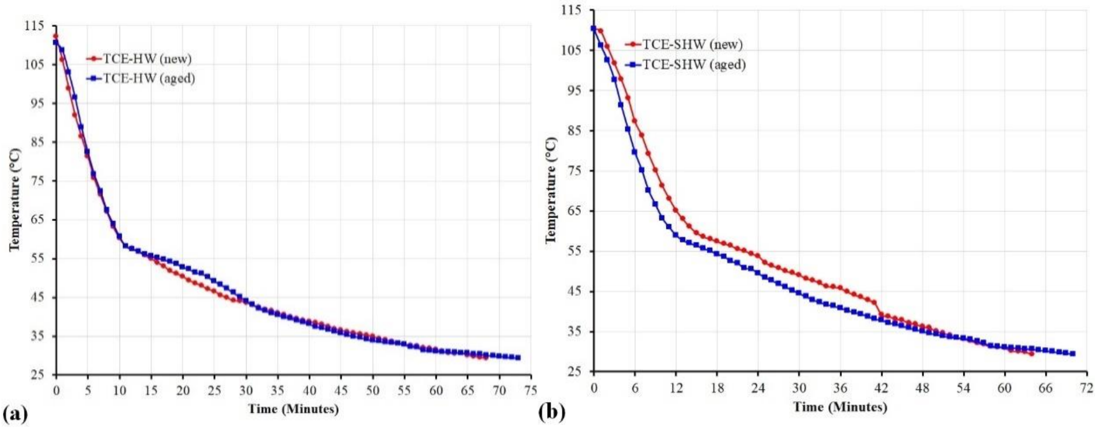

Figure 9. *Temperature releasement before and after aging for HW (a) and SHW (b)*

The solidification of hydrocarbon compounds is a complex process. Instead, phase stabilization with polymer to form TCE-PCM allows better distribution of solid additives. As a result, it is able to ensure the distribution of solid additives to help the heat releasement of the

PCM, particularly in the solid stage. It can be seen in the initial cooling stage after the solidus point, which is accelerated between 57.8°C – 42.1°C. Therefore, the presence of a phase stabilizer helps the designation of the operating system for the aged TCE-PCM. It ensures the long-term cycle of the TCE-PCM and improves the heat releasement process to ensure the stored energy can be discharged sufficiently.

4. Conclusion

Stabilized hydrocarbon wax (SHW) expresses a better performance to ensure the heating and cooling profile after liquidus aging. The addition of a polymer-based stabilizer maintains the chemical composition of HW. According to the XRD pattern, the presence of solid additives does not alter the phase and crystal structure of the PCM, implying a physical mixture without causing any drawbacks to the PCM. The microscopic profile of TCE-HW indicates the dendrite formation, which can be affected by the change in the solidification process for the hydrocarbon compound. It demonstrates that solid additives positively accelerate the heat transfer process within the HW.

The disruption after aging process clearly confirms the performance degradation of the TCE–PCM. It causes a slower melting rate during the heating stage for HW, around 22.5%, while SHW has a more minor decrement around 10.3%. It makes the SHW able to maintain the effective discharge rate while further adjustment is required for HW due to significant decrement on the average of melting rate. Moreover, the cooling and heating profile for SHW decreases unsubstantially compared to HW. Therefore, the phase stabilizer protects the PCM from severe degradation after treatment of liquidus aging. It ensures the operation of TES system can be maintained effectively by using SHW. The finding from this work can be taken as an important reference to ensure the optimal operation of hydrocarbon-based TES system which uses solid additives as heat transfer enrichment. Consequently, the reliability of the TES system can be obtained and ensure a stable operation.

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