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ORIGINAL STUDY

Preliminary Functionality Assessment on Supercooling Suppression of Binary Polyol Mixture as Energy Material for Thermal System

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Abstract

The functionality of the material has valuable aspect for energy utilization, specifically for optimization and renewable technology. Modification from the base material mostly performed to overcome some drawbacks to achieve the technical function, particularly for storing energy material (SEM). This work studies the modification of SEM-TS (thermal system) using polyols (sorbitol/SR and mannitol/MN) as the base SEM. Modification is performed to minimize the supercooling effect for the both polyols, and processed without causing fundamental changes on the structure and chemical composition. The mixture was produced at ratio 25 wt %, 50 wt % and 75 wt % (relative to SR), where the evaluation demonstrated various performance changes. The chemical identification shows the mixture has identic profile with the base, showing no chemical transformation. The diffraction from XRD is also similar for SR and MN, which also confirmed by the thermogravimetric curve which has identic decomposition pattern between the mixture and base material. Key improvement is achieved where the mixture indicates lower supercooling degree (5.1−10.8 °C). Also, each mixture indicate solidification transition occurs at longer range, showing the ability to recrystallize during discharge operation. Energy input/output evaluation demonstrate significant change related to temperature increment for heating cycle, giving a clearer indication on the change of melting mechanism of the mixture. It also observed for the heat discharge, showing the mixture has ability to releasing the stored heat in a better manner compared to SR. In general, functionality of the mixture as SEM-TS is more favorable than the base material, potentially unlocking the application of polyol-based SEM-TS in thermal system.

Keywords: Charging, Efficiency, Energy, Material, Polyol, Supercooling

1. Introduction

High technological improvement for the present day has a direct relation with continuous improvement on material aspect and its function. At the fundamental point, it focuses for supporting the energy transition, employing many different technology and advanced functional material [1]. Dissemination and distribution of an optimal energy model is achieved as the global society shifts to clean energy. Implementation of the technology, such wind turbine, is a clear milestone for the deployment of renewable system, proving research contribution and system optimization is extremely important [2−4]. It also applied for different field, including optimization for thermal system [5], which one of the largest energy consumption for the current society. Different effort is made to reduce the carbon footprint from this sector, mostly done by harvesting clear solar power to support the demand in thermal sector.

The successfulness for energy transition depends on the key component of the system, which is energy storage [6]. Specifically, the material that capable to store energy is another important achievement and evolve rapidly to meet the technical requirement. Thus, specific modification is required to overcome the

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insufficient fundamental functional of the material to meet the criteria as storing energy material (SEM) [7]. Many literatures report various modification, synthesize and characterization method for SEM in electrical battery. It also happens for thermal system (TS), making the advancement in SEM-TS growth rapidly. The key modification for SEM-TS mainly done by ensuring technical capability, including energy capacity, operation behavior and cyclic stability [8].

Numerous SEM-TS are available, where today attention focuses for possible exploration of renewable material, particularly from polyols group [9]. It has suitable characteristic from sustainable aspect, including thermodynamically favorable since it possible for operating temperature above 90 °C with high theoretical storage capacity than wax [10] and fatty acid [11]. It makes development is extensively addressed in this class for the given temperature operation [12]. Recent study showed polyol has more technical performance compared to molten salt and polyethylene [13]. Nevertheless, the base functional characteristic as SEM-TS is less feasible, making certain modification is demanded for polyol groups. At the basis, unsuitable cyclic behavior and metastable effect make the practical ability are insufficient, specifically for releasing the energy. Slow nucleation makes poor crystallization process, resulting in imperfect discharge and thermal exchange for the operation [14].

Technical consideration is made to solve the given challenge, focusing on system adjustment. For example, employing multi-tube arrangement for the operation SEM-TS made substantial operational advancement. It was marked by a higher specific power output, reaching 4.7 kW under suitable flowrate of the thermal transfer system [15]. Different adaptation was made by introducing passive operation as an effective approach by adding metal wool element. It produced to substantial contribution for the maximum power discharge that elevated to 205 % [16]. The SEM-TS operates based on fundamental concept of heat exchange through conduction and convection [17—19]. It made alternative modification for the component and the essential element of the thermal exchange for the SEM-TS increased the utilization factor to 71 % [20]. The advancement from previous work indicates a clear factor that need to be focused to solve the technical challenges and unfavorable operation of polyol as SEM-TS.

Field of material engineering is also contributed for the effort to improve the operational aspect of SEM-TS. The work [21] applied SEM-TS with additional feature of porous medium and combine it with nano additives. There was a strong relevance on the changes of operation duration as a direct result of the improved performance of modified SEM-TS. Strengthen the functional aspect of SEM-TS was applied using multi-layer for specific type application. It improved the operational consideration of the system and thermal distribution, making the efficiency enhanced to 68.2 % [22]. Specialized material such graphene and TiO_2 for the developed system improving the conductivity to 0.67 W/m·K, making it more favorable for thermal operation [23]. Despite the high achievement, one specific problem remain occurs crucially on the aspect of metastable and supercooling condition.

The high theoretical capacity of polyol group as SEM-TS is the key benefit. Unfortunately, the poor operational behavior occasionally found for this group due to high supercooling nature [24]. Effort to minimize it was done by adding ZnO, proven to reduce the supercooling to 6.5 °C with high performance indicator up to 98 % [25]. The assessment on the basic operational of the material indicated a suitable stability for 200 cycles with high latent heat of 284.3 J/g [26]. It makes modification may be performed by combining different polyols as alternative solution. For example, the eutectic mixture of group polyol has applied for various bio-based application [27]. The proposed approach is scrutinized suitable for SEM-TS that may changes the crystallization behavior and reduces the impact of supercooling [28]. Review work [29] recommends the mixing process of different polyol groups, whether forming eutectic mixture or not, could be achieved to maximizing the operation for SEM-TS.

The high potential for utilizing polyol as SEM-TS are attractive for further study. Previous work gives several recommendations, while a clearer result for the outcome of direct mixing with different polyol group is still missed. Thus, present study aims to answer the missing gap, preliminary working for assessing the mixture using functional assessment through assessment of various rating energy input and output for the functionality of polyol group as SEM-TS. The work is expected to provide additional reference regarding the interaction of energy-temperature for the mixture and its base, including the structural and chemical observation. It makes a detailed fundamental performance can be explored to understand the improvement on group polyol as SEM-TS.

2. Materials and method

The functional assessment was evaluated based on energy input and output for the produced samples. In this case, direct heating was performed as an excellent method for charge passively the SEM-TS where the intake energy is metered as electrical input [30]. Heater was located inside the test cylinder (SS 201, thickness 1.5 mm) which in touch directly with samples (Fig. 1a). The sample quantity (mass based) inside the cylinder

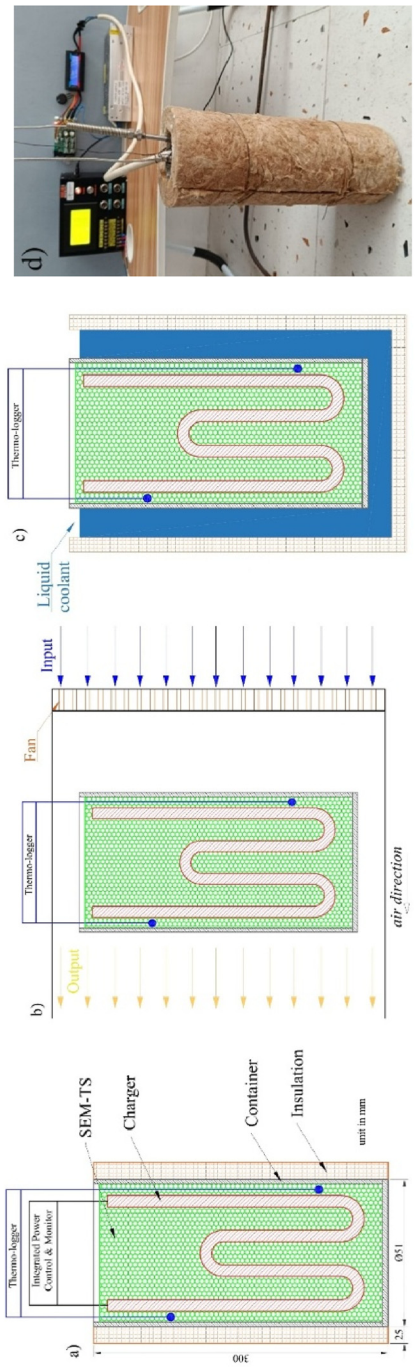

Fig. 1. Functional evaluation of SEM-TS for energy input (a), medium rating output (b), high rating output (c) and documentation of the process (d).

was 500 g for each test. There were two measurement points inside the chamber in order to observe temperature change of the sample throughout the operational assessment. The power for heating the samples were varied with three rating input (RI), which defined as low (LRI, 40 Watts), medium (MRI, 60 Watts), and high (HRI, 80 Watts). The purpose for the variation was to observe the melting behavior of the samples and analyze the impact of mixture between two polyol group at different ratio. The cylinder was insulated using glass wool to reduce potential heat loss. The upper temperature of the sample was 190 °C (maximum charging point).

The stored energy within the sample then released as a function of discharge. It was done through three variation model rating output (RO). Low RO (LRO) was set based free convection mode by removing the insulation and allow the air in contact with external surface of the cylinder. Medium RO (MRO) was performed as forced convection mode by air [31] which was supplied from blower (Fig. 1b). Finally, high RO (HRO) was used by placing the cylinder inside liquid coolant (water, Fig. 1c) which rapidly discharge the stored heat through the wall of cylinder [32]. Each test changes the heat transfer mode and consequently shifts the freezing mechanism, which is essential to observe the performance of the produced samples. In addition, the experiment documentation is shown in Fig. 1d.

The base material for this work was sorbitol (SR) and mannitol (MN) which came from polyol groups SEM-TS. It was obtained from local supplier with market grade category. The mixture was developed using three specific SR ratio relative to MN: 25 wt% (SMA), 50 wt% (SMB) and 75 wt% (SMC). The synthesized mixture was processed using direct mixing process by heating the mixture to achieve their liquid phase [33] at standard air temperature pressure. The base material was heated employing mechanical mixer at initial temperature 100 °C, and continue heated slowly until 200 °C. The proper dispersion mixture then taken for laboratory test for the phase structure using XRD (Malvern). The chemical composition was examined according to the Fourier-transform infrared (FTIR/PerkinElmer). Decomposition profile was evaluated using thermogravimetric method (NETZSCH) while surface observation was done using electron microscope (prisma-E) for base material and best sample from functional SEM-TS evaluation. The assessment was intended to support the analysis of functional evaluation.

3. Results and discussion

Basic operation for SEM-TS comes from the charge pattern, showing the relation of temperature increment in time. Varying the input energy impacts the charge characteristic which is observed based on the sample's temperature (Fig. 2). Also, the charge pattern is varied for each sample, showing there is specific intervention which comes from the tested material. It is the key consideration for modifying the SEM-TS in this work, showing each base material has significant operational behavior. MN generally shows steady temperature change for all RI. However, there is an alteration between 150 and 160 °C, which observed as small temperature drip for LRI (Fig. 2a) and slower temperature increment for MRI (Fig. 2b). The temperature is melting transition for MN [34], demonstrating the phase change alter temperature of the SEM-TS simultaneously. However, it is possible to minimize the issue by employing higher input energy (HRI) which may accelerating the melting transition and maintain the effective temperature increment for MN (Fig. 2c).

The charge pattern for SR is shown as rapid increment until 135 °C (Fig. 2a), which briefly disrupted before continue to increase until the designed target limit. However, the profile changes significantly using MRI (Fig. 2b) where there is clear change on charge pattern between 90 and 135 °C. It is the melting region for SR [35], indicating the phase behavior is only observed under suitable heating rate. It makes the functional phase transition is hardly to observe and may causes serious impact for charge protocol on actual system [36]. Adding MN to SR principally makes some changes on the temperature pattern, demonstrating there is certain modification on the melting behavior of the mixture. SMC demonstrates the most optimal profile for all RI scenarios, while clear implication is observed that phase transition occurs above 100 °C based on the shift variation. In addition, two stages temperature pattern is observed for SMA under LRI and MRI. It contributes significantly for operational SEM-TS system which makes temperature-time relation is possible to take as indication operation, including the supplied energy to the charger unit.

The temperature of the charge is used as determination of energy level since TS operates definitely under temperature differences. As result, the energy level-time profile is obtained for energy releasement of the samples (Fig. 3). The heat releasement is affected greatly by the solidification mechanism. For example, SR has rapid energy release during SD (Fig. 3a) and AD (Fig. 3b) operation, but it shows the opposite behavior under WD operation. SD and AD have the lowest convective coefficient while SR shows rapid temperature drop at this operation. It proves SR is unideal based on the key operation of SEM-TS. On the other hand, TS system may operate for discharge the heat

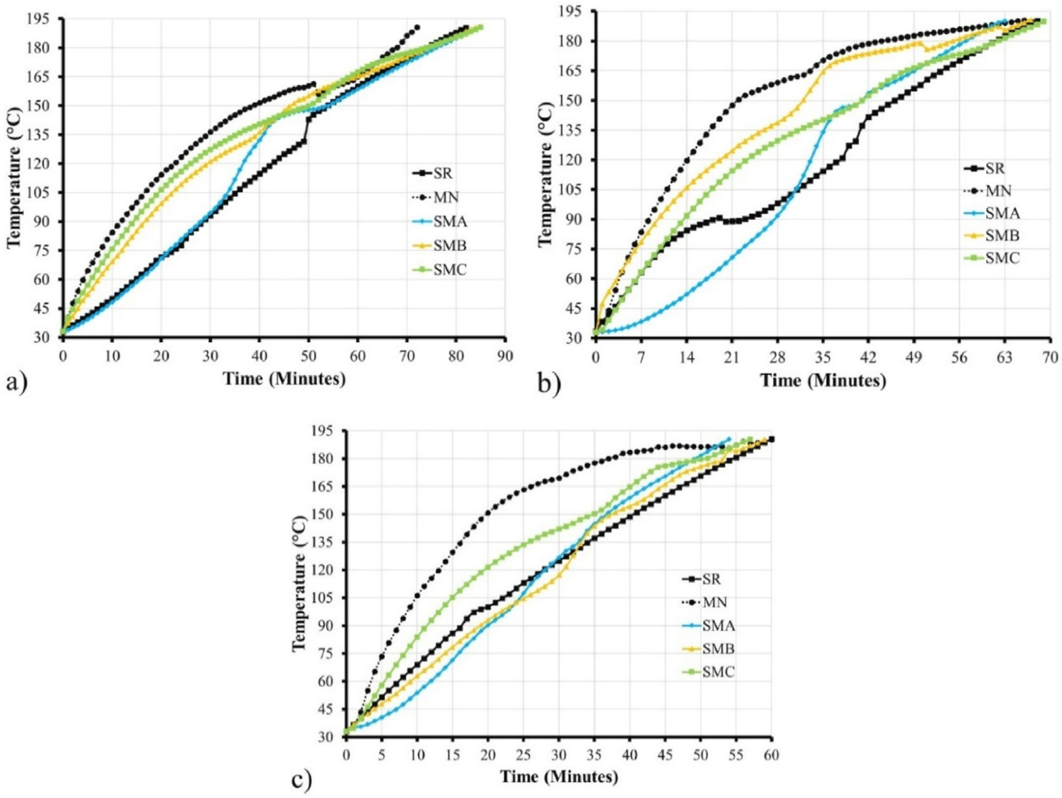

Fig. 2. Charge pattern for samples with LRI (a), MRI (b) and HRI (c) energy.

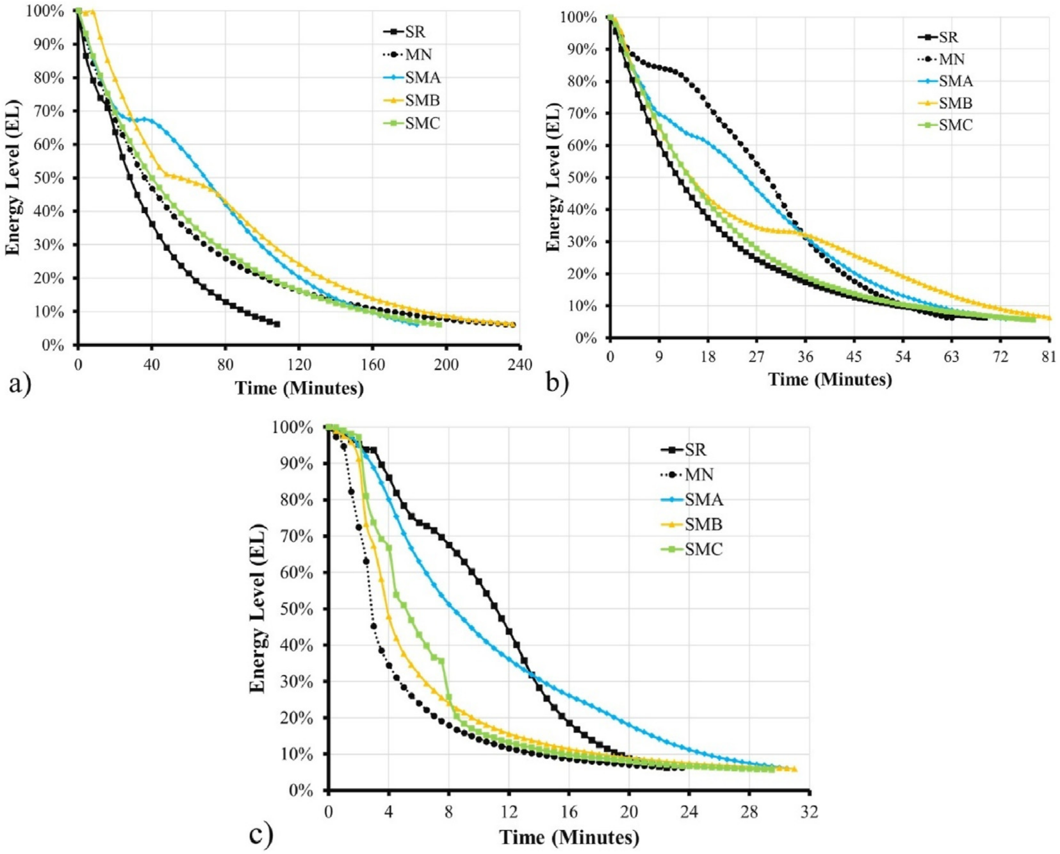

Fig. 3. Temperature release of the samples for SD (a), AD (b) and WD (c) operations.

using water, unfortunately SR has indication of slow heat discharge while the system demand rapid releasement. Thus, the imbalance causes significant impact for the actual TS operation. It is the main challenge for implementing SR as SEM-TS due to poor discharge performance due to supercooling nature.

Using MN shows the temperature drops steadily with SD operation (Fig. 3a). However, significant change is observed when forcing the air as discharge medium (AD, Fig. 3b) which indicates a slower energy level decrement between 90 % and 80 % (approximately 182.2—151.2 °C). It is associated with the latent heat releasement from the MN along with phase transformation. It also observed for all prepared mixtures which has MN content, specifically for SMA and SMB under SD and AD operation. Changing the discharge mode contribute the value variation, as observed for SMA with AD operation which occurs at 146.5—125.1 °C (70-60 %) and SD 145.2 °C—144.6 °C (68.4—65.6 %). In case of SMB, variation is shown between 100.2 °C and 82.9 °C (40-30 %) for AD and 113.2 °C—100.4 °C (50.1—47.3 %) for SD operation. Using WD makes the temperature drops rapidly which similar to MN profile (Fig. 3b), except for SMA which remain showing excellent decrement profile with steady behavior with less fluctuate profile. Thus, adjusting the MN ratio also plays significant benefit which only observed in fast energy release.

The change in discharge pattern (Fig. 3) specifically linked to the addition of MN on the SR. Additional assessment was taken to observed the phase structure from XRD characterization (Fig. 2). There is a clear remark that the obtained 2θ for SR and MN are identic with average deviation ±0.2°. It is observed for the given region at 11.6°, 18.6°, 20.3°, 21.8°, 22.5°, 23.3°, 25.4°, 28.9° and 33.7°. It implies the structural pattern for the both polyols (SR and MN) are coequal, emphasizing the identic process through hydrogenation for producing SR and MN while the key difference are the base carbohydrates. The interesting result is observed for the synthesized mixture between SR/MN. The main feature of the diffraction for each mixture is identic. Thus, additional examination is done for evaluating the chemical structure of the sample. It is shown in Fig. 5 as the presented version of infrared spectrum from FTIR. The primary constituent of the polyols produces similar spectrum at region 3.6—3 × 10^3 cm^{-1} with broad band curve that indicate OH— vibrations.

Another smaller intense profile is observed at 2.9×10^3 cm^{-1}, defined as the region of CH—stretching. The profile that shown in 1.4×10^3 cm^{-1} is related with vibrations of OH Bonds. At the given range, SR and MN has an identic spectrum region and profile. The main feature for each polyol occurs at 1×10^3 cm^{-1}, primarily indicating the key orientation of hydroxyl group between SR and MN. The synthesized mixture technically has identic pattern in the main region while the finger print shows correspond value of the MN ratio within the mixture. Thus, the primary chemical structure of the synthesized mixture is identic with base material without indicating any chemical changes.

Identification on the aspect of phase structure and chemical composite demonstrate the obtained sample have identic profile with the base material. However, according to the thermal properties, transition temperature varies notably for the produced sample (Fig. 6). At the base, there is no freezing point is observed for SR, showing the highest supercooling point. The condition is similarly occurred for MN, only at smaller value of 34.6 °C. Melting transition is indicated becomes longer for SMA, confirming the significant change in charge pattern under LRI (Fig. 2c) and MRI (Fig. 2b) operation. It shows there is certain changes on the phase behavior which alterns the energy interaction of the samples. Moreover, the important result is achieved as the mixture able to indicate lower supercooling degree than its base. It implies combination of the mixture may alter the crystallization behavior to maximize the solidification process. It is the main factor that contribute on the change of discharge behavior for all samples as observed in (Fig. 3). Despite that, solidification occurs at longer range compared to MN, showing the intervention of SR still occurred for the mixture.

Examination is continued to analyze the decomposition profile for the mixture to analyze the miscibility characteristic between SR and MN. According to Fig. 7, decomposition SR dan MN occur as direct process at 280—380 °C. It is the main issue for employing organic SEM-TS which has technical limitation for operating at higher temperature, especially when compared to inorganic salt that able to operate above 500 °C [37]. Still, the given temperature is excellent for operation of thermal system below 200 °C such residential heating and organic Rankine cycle, making the proposed model still relevant for operational thermal system in modern technology. In addition, all mixtures indicate the decomposition profile identic with the base material, including the occurrence at the same temperature span. It shows SR and MN are suitably mixed, confirming the XRD (Fig. 4) and FTIR (Fig. 5) pattern. Nevertheless, the indication should be further evaluated to assess the

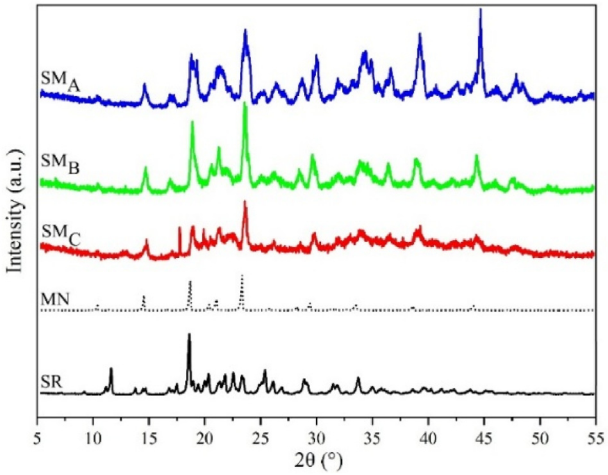

Fig. 4. The resulting diffraction pattern from the synthesized mixture and their base.

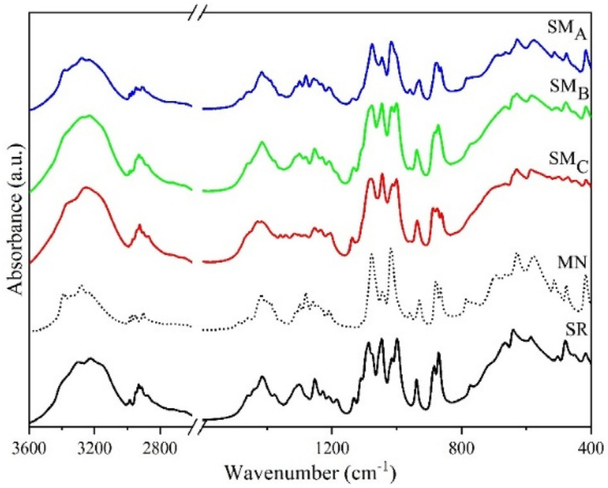

Fig. 5. The spectrum pattern for chemical evaluation using FTIR.

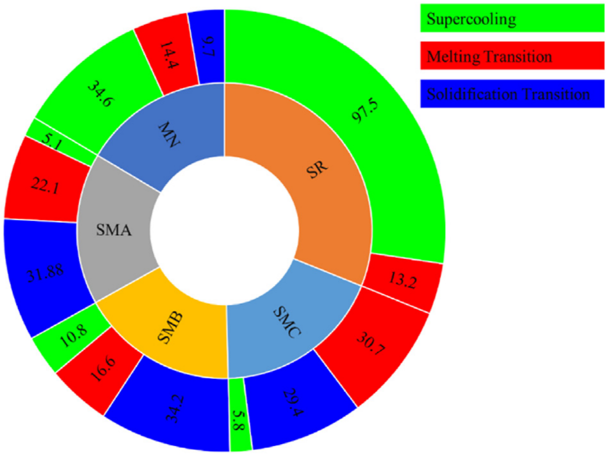

Fig. 6. Variation on the specific temperature phase transition and supercooling.

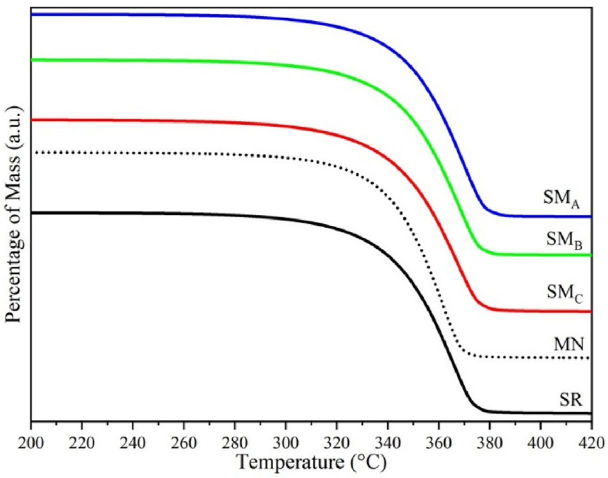

Fig. 7. Decomposition profile of the assessed samples.

mixture for possible long term operation through thermal endurance test [38] since the mixture probably experience some deterioration due to aging process that may alter its performance as SEM-TS.

The operational characteristic of SEM-TS depends highly on the average rating input energy, which correlates with average temperature increment of the material. As plotted in Fig. 8a, each sample shows significant variation on the average rating input. Specifically, the basic performance of MN on LRI operation has higher input than SR around 14.1 %. The energy input pattern for SR (Fig. 2a) causes the all mixtures has lower value. Increment on the melting transition range (Fig. 6) affects it, which makes the energy input occurs slowly. It is possible to be solved by adding conductivity enhancer, especially nano-based material [39]. Oppositely, increasing the RI allows the mixture to achieve a suitable input behavior, especially for SMA under MRI and HRI operations. SMA has optimal rating input with specific value of 2.49 °C/min and 2.91 °C/min. It is one significant benefit since the charge can be accelerated and allow the thermal response of the system to be enhanced.

One critical trouble for MN is the average heat loss behavior which generally faster than SR (Fig. 8b). It becomes clearer for the mixture as the MN content becomes higher, specifically for SMB and SMC, which has identic value around 3.75–3.72 °C/min. The finding requires additional consideration since it shows the material has higher self-discharge and demand sufficient insulation to prevent from the condition. In contrast, the ratio of the mixture also causes the change, as observed for SMA, it has lower value for all scenario RO operation. It confirms the temperature transition during solidification (Fig. 6) for SMA (31.88 °C) which is smaller than SMB (34.2 °C) and higher than SMC (29.4 °C). Thus, key consideration on the functionality of the mixture as SEM-TS is achieved on SMA.

Further assessment was considered using microscope observation between the base material (SR and MN) and best mixture (SMA). MN indicates clear crystal pattern which scattered in each region while some part indicates larger crystal profile (white arrow Fig. 9a). It proves MN able to recrystallize despite the high supercooling occurred (34.6 °C). The supercooling causes some of the solidified crystal agglomerated in one region (green arrow), demonstrating poor solidification mechanism which causes low heat rejection rate (Fig. 8b) when using low rating output. Despite that, SR shows clear boundary layer (red arrow Fig. 9b) which implies that the nucleation occurs slowly, causing each region might remain in liquid region after extended duration. It results in severe temperature drops for SR (Fig. 3), which becomes accelerated once the rating output is designed for fast discharge cycle (Fig. 8b).

Functional evaluation between the SR-MN mixture as SEM-TS is observed better as the mixture indicates sufficient blending profile. It is indicated by smooth

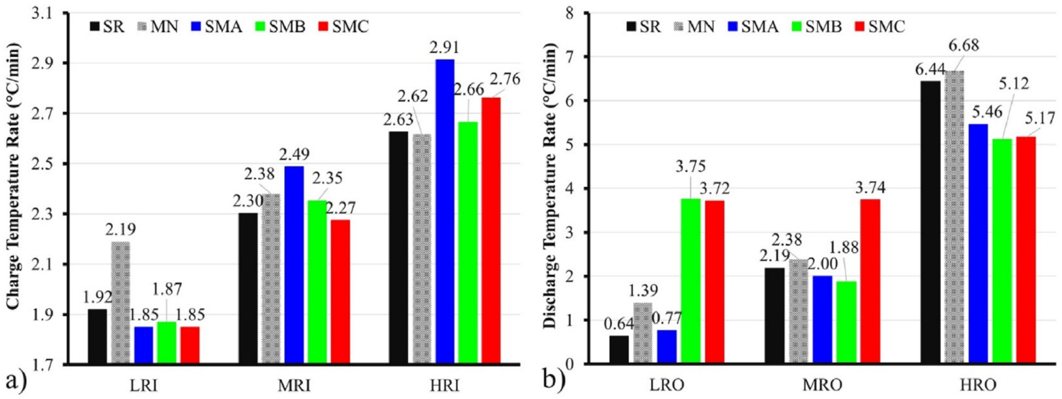

Fig. 8. Comparison for the average energy input (a) and output (b) for the samples.

layer formation which extended in the surface without indicting any agglomeration or clear crystal pattern (Fig. 9c). It proves the XRD (Fig. 4) and FTIR (Fig. 5) pattern for the mixture which remain identic with the base material. Moreover, it emphasizes the similar decomposition profile of the mixture and base material as plotted in Fig. 7. However, electron microscope profile indicates some void region (blue arrow) and stacked layer (yellow arrow) which indicates the quality of the mixture requires additional modification to ensure perfect formation without causing any black region. Despite that, the key functionality of SMA is shown as ideal performance compared to SR and MN, making the mixture is preferable for application of SEM-TS in thermal system. Still, further modification is recommended to maximize the finding, including the possibility for employing thermal enhancer and thermal cycling test to understand long term operation and suitability for the mixture as SEM-TS.

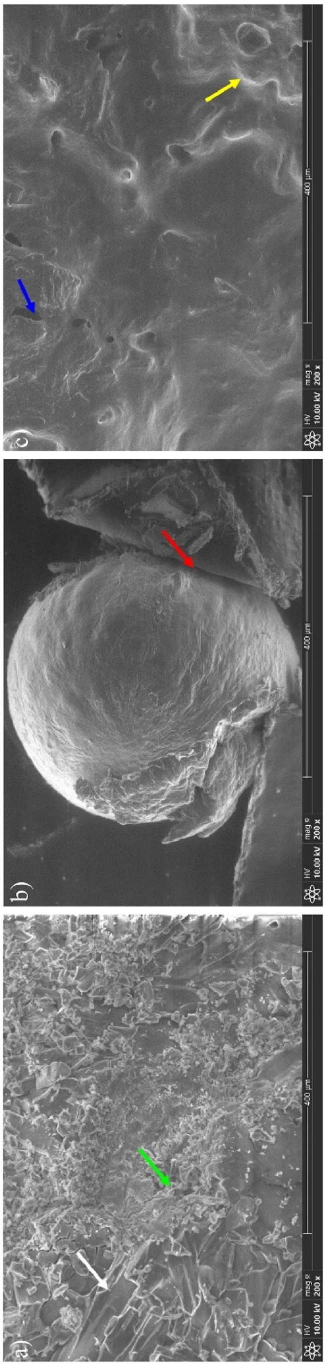

Fig. 9. Electron microscope profile for MN (a), SR (b) and SMA (c).

4. Conclusion

Preliminary assessment for improving the functionality of polyol as storing energy material (SEM) in thermal system (TS) is done by evaluating the charge/discharge pattern of sorbitol (SR), mannitol (MN) and their mixtures (SM). The SR is designed as base polyol and demonstrate the poor operational behavior from the perspective operation of SEM-TS. It is obtained according to the supercooling value, followed by charge rate which considerably insufficient compared to MN. In other hand, MN also suffers from insufficient discharge and indicating the supercooling point around 34.6 °C. Microscope observation confirms the high agglomerated profile (SR) and scattered crystal distribution (MN), which highly correlated with the poor nucleation rate. Combining the SR and MN indicate various improvement, which specifically can be observed based on the decrement in supercooling for the mixture that has lower value than the base materials. Microscope profile for the mixture demonstrates excellent miscibility, confirming the XRD, FTIR and TGA profile which has identic indicator between the mixture and base material.

The finding shows the key improvement on the charge/discharge rate for the mixture, followed by improvement on the solidification rate. Nevertheless, the evaluation focused on basic analysis for developing polyol-based SEM-TS. Thus, advance studies are required by focusing the following topics:

- Cyclic test and thermal endurance assessment to assess long term operation of the mixture, including the possibility for phase segregation that may occur after long cycle operation.

- Conductivity enrichment for improving the heat transfer rate of the SEM-TS, ensuring high thermal response which is crucial for the operation of the system's operation.
- Operational modification and adding secondary binder material to improve both the crystallization kinetic and overall performance as SEM-TS.

We believe the finding is essential to achieve advance SEM-TS, making the proposed model is applicable for actual system and help the achievement of higher operational function of the SEM-TS.

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Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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