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Salt Hydrate Eutectic Mixtures for Near-ambient Thermal Energy Storage Applications

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ABSTRACT

In this work, we synthesized and characterized Na₂SO₄·10H₂O – Na₂CO₃·10H₂O eutectic salt hydrate phase change material (PCM) using Differential Scanning Calorimetry (DSC), Attenuated total reflectance infrared spectroscopy (ATR-IR), and X-ray Diffraction (XRD) techniques. The eutectic point of the PCM mixture was predicted based on binary eutectic effect and ion exchange theory. The XRD and ATR-IR results indicate that the eutectic PCM mixture was formed based on only physical mixing without chemical interaction. Moreover, DSC results showed that the eutectic concentration is 50wt% Na₂SO₄·10H₂O – 50wt% Na₂CO₃·10H₂O with melting temperature of 25.18°C and melting enthalpy of 187.46 J/g. Also, 78% of melting enthalpy was recoverable after 10 melt/freeze cycles. This eutectic mixture has the potential to be used in building thermal comfort applications.

1. INTRODUCTION

The rising utilization of renewable energy sources has enhanced the need for energy storage technologies. One of the notable energy storage solutions is thermal energy storage (TES) in which systems store and release thermal energy for a variety of applications (Khan et al., 2016). Building systems, space heating and cooling, and refrigeration are near ambient temperature applications where TES systems can thrive. Latent heat thermal energy storage (LHTES) is a very efficient method of storing energy. LHTES entails the use of phase change materials (PCMs) in thermal systems and devices to store and release energy (Sharma et al., 2009). In addition to energy storage, PCMs have been shown to be useful in cooling, desalination, and heat exchangers. Thermally sensitive systems, in which big temperature variations are undesirable, are where they come into play. The energy efficiency, adaptability, and load reduction are all improved as a result of the application of LHTES (Kumar et al., 2019).

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However, there are significant drawbacks to using salt hydrate PCMs, including low thermal conductivity, erratic supercooling, incongruent melting leading to phase separation, and low volumetric energy storage capacity (Purohit & Sistla, 2021)(Goswami et al., 2021). These drawbacks have been studied and several solutions proposed to improve salt hydrate PCM thermal performance. This comprises thickeners (Cong et al., 2022), nucleators (Kalidasan et al., 2022), and encapsulating materials (Nazir et al., 2019). Another new strategy involves mixing two or more salt hydrate PCMs in a specified concentration to generate eutectic mixes with low supercooling and congruent melting.

Several studies have been published on the formulation and synthesis of eutectic salt hydrate mixtures for use at 25–30°C. For instance, Fang *et al.* (2020) developed a eutectic PCM composite of 20wt% $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ and 80wt% $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$. 2.5 wt% $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ was added as nucleator and 30wt% SiO_2 as shape stabilizer. The resulting PCM composite has phase change temperature of 25.16°C, melting enthalpy of 142.9J/g and negligible supercooling. In another work, Xie *et al.* (2019) reported a shape-stabilized PCM composite made by impregnating expanded vermiculite with a 1:1 mass ratio of $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ and $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$, yielding a PCM composite with a 60 wt% salt hydrate. The latent heat and melting point of the PCM composite were 110.3 J/g and 23.98°C, respectively. Also, Li *et al.* (2014) reported a eutectic PCM mixture of 75wt% $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ and 25wt% $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$. The nucleators used were 3wt% SrCl_2 and 1wt% SrCO_3 , and the thickener used was 0.5 wt% hydroxyethyl cellulose. The PCM composite has a melting temperature of 21.41°C and a melting enthalpy of 102.3 J/g.

Li *et al.* (2011) recommended that PCMs be used in buildings with a phase change temperature of 18–22°C in the wintertime and 22–26°C in the summertime. However, majority of the investigations found eutectic PCMs with lower melting enthalpy or phase transition temperature beyond the range of indoor thermal comfort in building applications. Also, there is a variation in the eutectic concentrations of the same PCM constituents reported in the literature. Hence, there is a need to further validate the true eutectic concentration of PCM mixtures for better materials design.

In this work, we developed a eutectic salt hydrate PCM mixture using $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ and $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$. The thermal characteristics of PCM composites were examined using differential scanning calorimetry (DSC). The crystal structure and chemical structure of the samples were investigated using X-ray diffraction (XRD) and attenuated total reflectance infrared spectroscopy (ATR-IR). This work is significant as it provides a eutectic PCM mixture with optimum properties for applications such as human comfort cooling and heat pump integration.

2. MATERIALS AND METHODS

2.1 Materials Synthesis

The inorganic salt hydrate PCMs utilized in this study were: $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ (SSD, purity>99 percent), and $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ (SCD, purity>99 percent). These reagents were purchased from Sigma Aldrich and were used in their original form without further purification. The materials were handled only in a humidity- and temperature-controlled glovebox to prevent water loss from salt hydrates.

The binary salt hydrates (BSH) were prepared by mixing SSD and SCD using the heat-mixing method (Rao et al., 2018). To begin, a borosilicate vial was filled with respective concentrations of SSD and SCD. It was then immersed in the water bath of 60°C and stirred for one hour within the water bath to ensure complete melting as shown in Figure 1. Afterwards, these PCM mixtures were allowed to spontaneously cool down to room temperature and the resulting BSH mixtures were obtained.

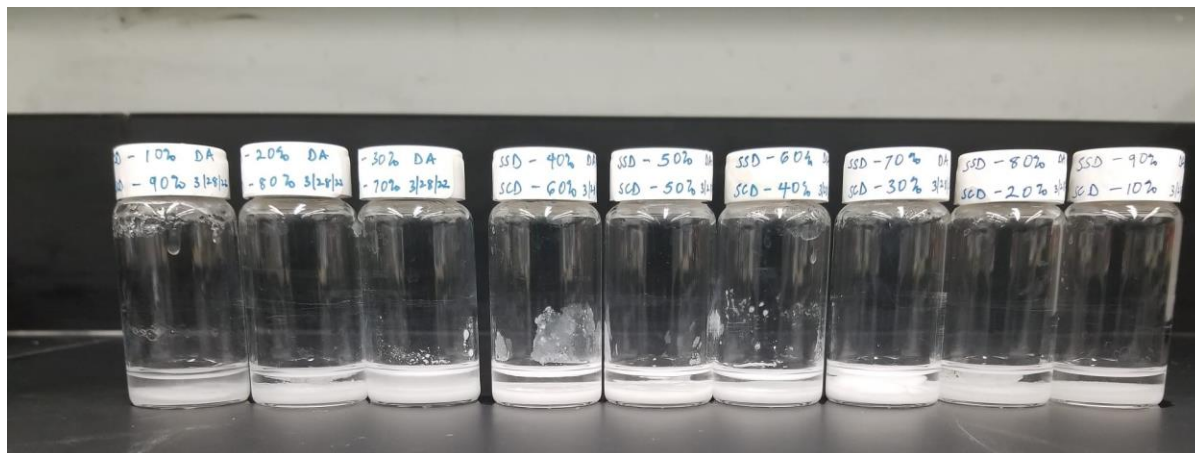


Figure 1: PCM mixture of sodium sulfate decahydrate and sodium carbonate decahydrate in various concentration in the melted state

2.2 Characterization

Differential scanning calorimetry (TA Instruments, DSC 2500) was used to measure sample thermal properties such as phase transition temperature and melting enthalpy between 5 and 45 degrees Celsius at a scanning rate of $2^{\circ}\text{C}\cdot\text{min}^{-1}$ under nitrogen at a flow rate of $50\text{ mL}\cdot\text{min}^{-1}$. About 10-15 mg of sample was used in each experiment. For each experiment, an analytical balance (Mettler Toledo, Model) with an accuracy of 0.0001 mg was used to measure the sample sealed in the hermetic DSC pan.

To determine the crystal structure of the samples, the X-ray diffraction patterns of the salt hydrate samples were analyzed employing the PANalytical Empyrean XRD diffractometer. The X-ray voltage was 45 kV and tube current 40 mA, respectively, with a Cu anode with a K_{α} of 1.54 \AA . The scan was performed at one scan per minute at 2θ angle ranging from 5 to 90 degrees.

To characterize the chemical structure and surface chemistry of the samples, the attenuated total reflectance infrared (ATR-IR) spectra of the samples were acquired using a PerkinElmer Frontier spectrometer equipped with a diamond ATR attachment with a spectral resolution of 2 cm^{-1} over the $4000\text{--}600\text{ cm}^{-1}$ range.

3. RESULTS AND DISCUSSION

3.1 Determination of the eutectic point

To determine the optimal concentration and the thermal properties of the PCM mixtures, DSC melting data of the mixtures are shown in Figure 2. Two endothermic peaks are observed in most of the curves. This indicates that there are multiple phases in the mixtures. The peak at the higher temperature range could be due to the melting of the mixtures while the second endothermic peaks at the lower temperature range is due to the eutectic effect (Liu & Yang, 2017). Only the DSC melting curve obtained for a mass proportion of 50% SCD (BSH5) presented a single peak in these mixes.

Also, it is important to note that as the mass proportion of SCD increases, the DSC peaks at higher temperatures gradually move to lower temperatures, eventually producing a single peak with an estimated melting temperature of 25.18°C and latent heat of 187.46 J/g (Table 1). The melting temperature of the mixture makes it a beneficial material for near-ambient thermal energy storage applications (Li *et al.*, 2011). Furthermore, the thermal properties of these PCM were investigated to show their melting behavior. The phase diagram of the PCM mixture is shown in Figure 3 which depicts the exact form for a binary mixture with eutectic point. In light of the above discussion, it

may be concluded that the mixture becomes a eutectic as the weight percentage of SCD approaches 50wt%. This eutectic formation of the salt hydrate mixture is predicated on the equivalent exchange of dissociated ions from each participating salts in mixed water (Watanabe & Hirasawa, 2018). The salt hydrate will be stable at the eutectic concentration due to the constant numerical relationship maintained by the participating ions. Here the cation, Na^+ is common between the two salt hydrates, it is not considered in the participation. Hence, the anions, SO_4^{2-} and CO_3^{2-} exist in a numerical relationship of 1:1 to ensure the sustenance of the eutectic point. A range below or above this ratio leads to severe precipitation of the anhydrous salt ions from the mixture, thereby producing a mixture with more than one phase.

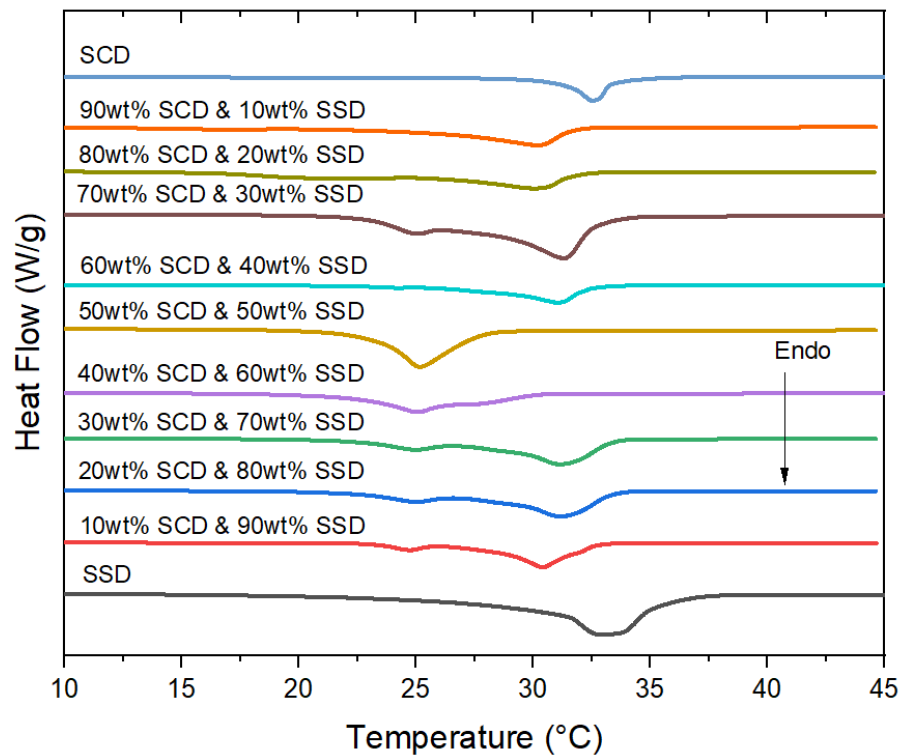


Figure 2: DSC melting curves of $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ (SSD), $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ (SCD), and their mixtures. Sample with 50wt% SCD (BSH5) showed a single melting peak indicating a sharp melting point

Table 1: Composition and thermal properties of the PCM mixtures

Sample	mSSD: mSCD	T_1 (°C)	T_2 (°C)	Melting Enthalpy (J/g)
SSD	100:0	-	32.94	145.87
BSH1	90:10	24.68	31.16	121.03
BSH2	80:20	24.87	30.41	181.88
BSH3	70:30	25.91	29.82	136.17
BSH4	60:40	25.20	27.82	126.23
BSH5	50:50	25.18	-	187.46
BSH6	40:60	25.12	27.17	142.36
BSH7	30:70	25.07	29.19	124.22
BSH8	20:80	24.29	30.47	170.71
BSH9	10:90	17.84	31.22	146.86
SCD	0:100	-	32.55	135.613

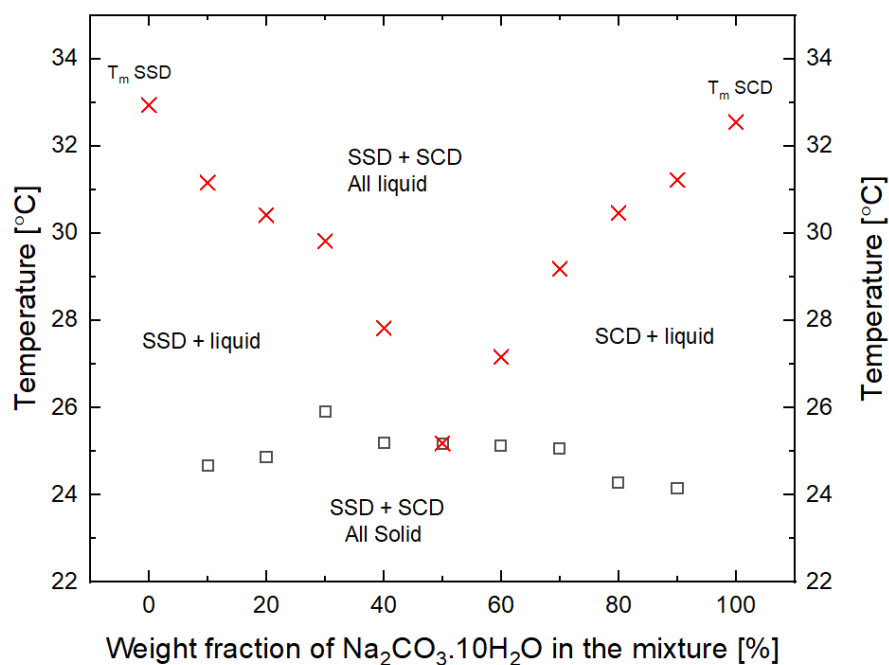


Figure 3: Phase Diagram of SCD+SSD, □ represents melting temperature associated with the eutectic point

3.2 Crystal Structure Characterization

The XRD pattern of pure SSD, SCD and the eutectic salt hydrate, BSH5 are shown in Figure 4. Pure SSD showed multiple sharp peaks with positions at $2\theta = 16.2^\circ, 18.6^\circ, 23.2^\circ, 26.2^\circ, 27.9^\circ, 31.5^\circ, 35.7^\circ,$ and 43.1° . These peak positions agree with already published crystal structure in the literature (Levy & Lisensky, 1978). Also, the characteristic peak of SCD are at the positions with $2\theta = 16.6^\circ, 19.7^\circ, 22.5^\circ, 28.4^\circ, 29.6^\circ, 30.9^\circ, 33^\circ, 37.8^\circ,$ and 41.1° (de Wolff, 1957). Furthermore, the eutectic mixture, BSH5 showed multiple overlapping peaks with the diffraction peaks of SSD and SCD present in the pattern as shown by the indexing. This confirms that the eutectic mixture, BSH5 was formed by the physical mixing or overlay of SCD and SSD salt hydrates without any occurrence of chemical reaction since no new product was formed.

3.3 Chemical Structure Characterization

Figure 5 depicts the ATR-IR spectra of the samples. The peaks at 1671 cm^{-1} and 3335 cm^{-1} in the spectra of pure SSD were ascribed to the O-H stretching vibration. the distinctive symmetric and asymmetric stretching vibrations of S=O in O-SO₂-O were at 1084 cm^{-1} and 1453 cm^{-1} , while the bending peak of S-O of SO₄²⁻ in SSD was assigned to the peak at 615 cm^{-1} . This further confirms the tetrahedral configuration of SO₄²⁻ in SSD (Liu et al., 2020). For pure SCD, the peaks at 636 cm^{-1} and 1103 cm^{-1} corresponded to the in-plane and out-of-plane bending vibrations of CO₃²⁻, respectively. At 1373 cm^{-1} , the peak related to the asymmetric vibration of CO₃²⁻ (Rao et al., 2018). The characteristic peak at 1671 cm^{-1} suggests the different vibration angle of O-H in H₂O. Also, the broad absorption peak at 3335 cm^{-1} represents the stretching vibration of O-H in H₂O. The broadness of the peak between $3000 - 3600\text{ cm}^{-1}$ could be associated to the hydrogen bonding in the water of crystallization. The characteristic O-H stretching band at a lower frequency associated with the compound is widened by hydrogen bonding, which has an effect on the infrared O-H spectra of materials. (Yazdani et al., 2021). Furthermore, the spectra of BSH5

showed no additional absorption peak, indicating that there was no chemical interaction between the constituents of the eutectic mixtures. This further corroborated the XRD results above.

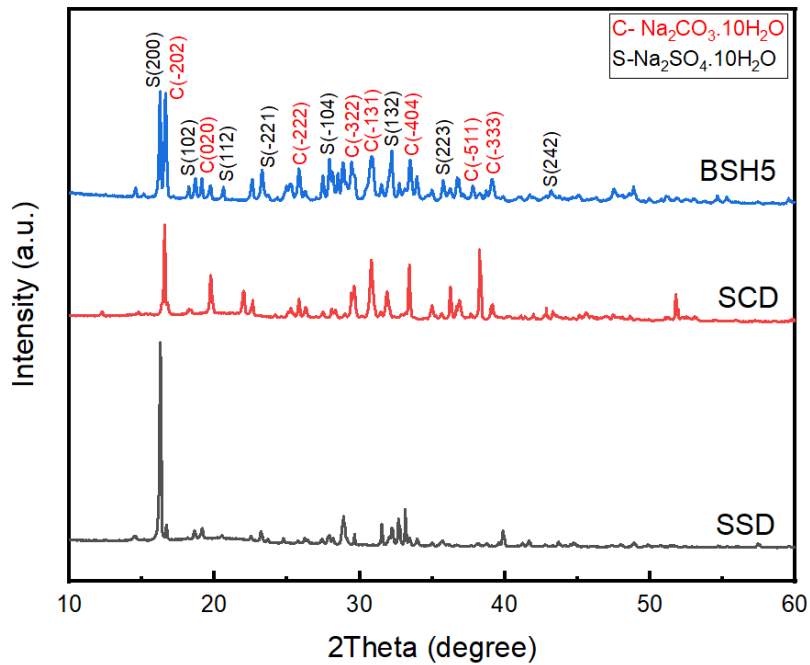


Figure 4: XRD patterns of pure SSD, SCD, and BSH5.

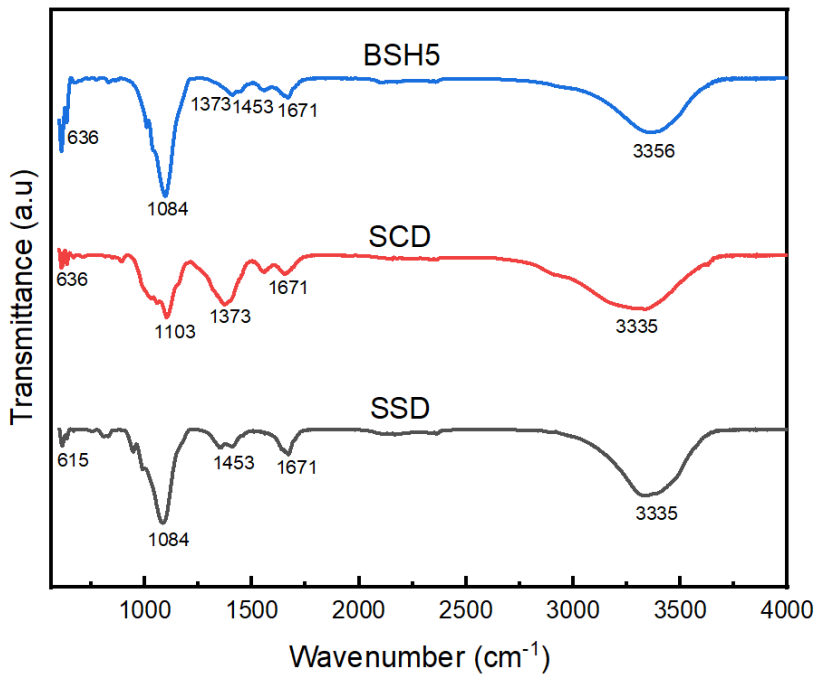


Figure 5: ATR-IR spectra of pure SSD, SCD, and BSH5. BSH5 is the eutectic mixture of SSD and SCD

3.4 Thermal Cyclic Stability of the Eutectic Mixture

Thermal stability is a key selection requirement for PCM mixtures used in thermal energy storage. The thermophysical characteristics of the PCM must remain stable when tested in multiple cycles of melting and freezing. Hence, the thermal cycling test was conducted on BSH5 using DSC analysis which enabled the evaluation of melting enthalpy and phase change temperature for 10 cycles. The thermal cycling curves of BSH5 are illustrated in Figure 6. It was observed that the melting enthalpy of BSH5 decreased after each successive cycle. According to Table 2, the melting enthalpy at cycle 1 was 189.42 J/g, and the melting enthalpy after 10 cycles was 154.43 J/g with enthalpy loss percentage calculated to be 22% after 10 cycles. Also, the phase change temperature of the eutectic mixture was observed to vary by 0.85°C during the thermal cycling experiments. This could be assumed to be negligible. Hence, the eutectic mixture, BSH5 exhibits sharp and consistent melting point under repeated thermal cycles making it suitable for thermally sensitive systems and applications requiring storage materials with consistent melting temperature.

To further enhance the thermal properties of the eutectic PCM mixtures for long-term applications, other viable methods and approaches could be adopted. Some of these methods to adopt include the use of polymeric or highly viscous materials which forms a network structure or physically thickened mixture thereby limiting the phase separation (Fang et al., 2021) or polyelectrolyte stabilization (Li et al., 2022) which involves the use of polyelectrolytes to provide electrostatic and steric effects which stabilizes the PCM mixtures.

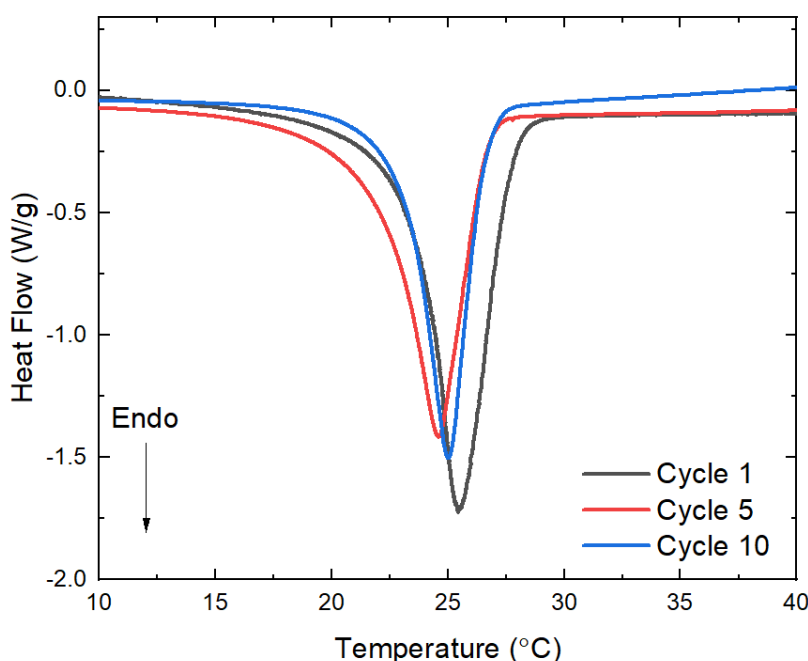


Figure 6: Melting enthalpy curves showing thermal cyclic stability test for BSH5

Table 2: Thermal cycling data for SSD-SCD Eutectic PCM mixture

Cycle	Onset of Melting (°C)	Melting Point (°C)	Melting Enthalpy (J/g)	Enthalpy loss percent (%)
1	23.38	25.45	189.42	-
5	21.93	24.59	162.52	14
10	22.92	25.02	154.43	22

4. CONCLUSION

Herein, we investigated the potential of PCM mixtures of $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ and $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ as new eutectic binary salt hydrate (BSH) PCMs for use in indoor thermal comfort in buildings systems. This mixture is formed by mixing $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ and $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ with a mass ratio of 50:50, which has a melting temperature of 25.18°C and melting enthalpy of 187.46 J/g . Also, 78% of the latent heat was recovered from the eutectic mixture after 10 successive melt/freezing cycles. The results of ATR-IR and XRD experiments reveal that the salts $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ and $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ in the eutectic system are involved in a physical interaction that results in the eutectic effect with lower melting point. Overall, the eutectic PCM mixture is a beneficial storage material for application in temperature control systems.

NOMENCLATURE

ATR-IR	Attenuated total reflectance infrared spectroscopy
BSH	Binary salt hydrates
DSC	Differential scanning calorimetry
LHTES	Latent heat thermal energy storage
PCM	Phase change materials
SCD	Sodium carbonate decahydrate
SSD	Sodium sulfate decahydrate
TES	Thermal energy storage
XRD	X-ray diffraction

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