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Experimental Investigation of Specific Heat Capacity of Ternary Nitrate Salt Mixtures for Thermal Energy Storage System in Concentrated Solar Power System

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Abstract

Concentrated solar power (CSP) systems require efficient thermal energy storage (TES) materials to address the intermittent nature of solar radiation. This study investigates the specific heat capacity (C_p) of novel ternary nitrate salt mixtures composed of potassium nitrate (KNO_3), lithium nitrate ($LiNO_3$), and magnesium nitrate hexahydrate ($Mg(NO_3)_2{\cdot}6H_2O$) for TES applications. Seven compositions were prepared and characterized using differential scanning calorimetry (DSC) over a temperature range of 30°C to 400°C. The results reveal that the specific heat capacity of all mixtures increases with temperature, ranging from 0.5845 J/g-K to 0.9987 J/g-K at 375 K, with the 40% KNO_3, 30% $LiNO_3$, and 30% $Mg(NO_3)_2{\cdot}6H_2O$ mixture exhibiting the highest C_p value of 0.9987 J/g-K. This enhancement is attributed to increased ionic mobility and thermal vibration at elevated temperatures. All mixtures exceed the minimum C_p threshold of 0.5 J/g-K required for TES applications, demonstrating their suitability for CSP systems. The findings highlight the potential of these ternary nitrate mixtures to improve energy storage efficiency, reduce operational costs, and support the broader adoption of CSP technologies. This study contributes to the development of advanced molten salt mixtures for next-generation TES systems, offering a pathway toward more sustainable and efficient renewable energy solutions.

1.0. Introduction

Solar energy has emerged as a leading source of renewable energy worldwide, primarily due to its clean nature, abundant availability, and ease of harnessing [1]. The global energy crisis can be minimized by utilizing the possible available solar energy. One of the efficient ways of utilizing solar energy is the use of concentrating solar power (CSP) technologies, which capture and convert radiation of the sun into useful heat energy for various uses such as thermal processing and electricity [2 - 4]. The characteristics of CSP have made its application gain attention globally over the past decade. However, the stable and continuous operation of CSP face challenges from the intermittent and volatility of sunlight. To address the mentioned problems in CSP a thermal energy storage is normally incorporated into the concentration-collection-generation process [5]. Alva et al. [6] and He et al. [7] stated that thermal energy storage has become very important in the establishment of CSP because of its security, high efficiency, reliability and flexibility.

The importance of thermal energy storage materials has been established in CSP applications [8, 9]. In operating the CSP heat storage, the thermal energy storage materials should possess some major features such as low cost, low flammability, good thermal stability, high thermal conductivity, and

high specific heat capacity for good heat storage and transfer ability [10]. This has made it imperative to determine these thermal properties of selected molten salts to know if they meet the specification for heat storage application in CSP plants. One of the vital thermal properties of molten salt is its specific heat capacity. Specific heat capacity is the amount of heat energy required to raise the temperature of a unit mass of a substance by one degree Celsius (or Kelvin) [11]. Analyzing the specific heat capacity curve provides valuable insights into the thermal properties of a substance, enabling optimization of its performance in applications like thermal energy storage. The overheating and start-up heat for molten nitrate salt when used as heat transfer fluid in solar energy storage applications can be controlled by knowing the specific heat capacity value of the salt [12]. Peng et al. [13]; Sang & Liu [14] also reiterates the importance of determining the specific heat capacity of molten salts, stating that an increase in specific heat capacity increases the available thermal energy of the molten salt, resulting in higher overall efficiency and lower generation costs in CSP solar energy applications.

Nitrate salt mixtures are widely used as phase change materials (PCMs) for thermal energy storage (TES) due to their high specific heat capacity, stability, and cost-effectiveness. Common nitrate salt mixtures such as Sodium and Potassium Nitrates ($NaNO_3$-KNO_3 and $NaNO_3$-$NaNO_2$) have been widely used binary salt mixtures with a phase change temperature around 230°C and ternary salt mixture exhibiting a phase change temperature around 270°C respectively [15]. Previous studies have revealed that nitrate salt mixtures are environmentally friendly, that is they are non-corrosive and non-toxic [16]. Based on these advantages nitrates salts mixtures offer a promising solution for thermal energy storage. These have led to numerous studies to have investigated the specific heat capacity of nitrate salt mixtures for thermal energy storage. Such studies are Wu et al. [17] that determine the specific heat capacity of a 50:50 wt% $NaNO_3$-KNO_3 mixture and Sergeev et al. [18] also evaluated the specific heat capacity a 60:40 wt% $NaNO_3$-$Ca(NO_3)_2$ mixture for binary nitrate salt mixtures. Similarly for ternary nitrate mixtures, studies by Wang et al. [19] and Delise et al. [20] determined specific heat capacity values for a 40:30:30 wt% $NaNO_3$-KNO3-$Ca(NO_3)_2$ mixture and a 50:20:30 wt% $NaNO_3$-$NaNO_2$-KNO_3 mixture respectively. Also, Redzuan [21] determined specific heat capacity of a 30:20:20:30 wt% NaNO3-KNO_3-$Ca(NO_3)_2$-$LiNO_3$ mixture for quaternary nitrate salt mixtures. These studies demonstrate the potential of investigating different nitrate salt mixtures specific heat capacities for thermal energy storage. Thus, it is evident that the determination of specific heat capacity for new nitrate salts mixture is necessary to know it suitability for TES application.

Kwasi-Effah et al. [22] developed a novel ternary nitrate mixtures for Thermal Energy Storage application. The novel ternary nitrate salt mixtures developed consists of potassium nitrate (KNO_3), lithium nitrate ($LiNO_3$), and magnesium nitrate hexahydrate ($Mg(NO_3)_2{\cdot}6\ H_2O$). The study carried out the thermal conductivity of the novel nitrate mixtures for its seven compositions. The study concluded that the nitrate mixtures were suitable for TES, as evidenced by their thermal conductivity values. However, to further confirm their suitability, it is recommended that their specific heat capacity, thermal stability, and viscosity be determined. Arising from the paper suggestion, it imperative to determine the specific heat capacity of the novel ternary nitrate salt mixtures developed consists of potassium nitrate (KNO_3), lithium nitrate ($LiNO_3$), and magnesium nitrate hexahydrate ($Mg(NO_3)_2{\cdot}6\ H_2O$).

This study aims to advance scientific knowledge on novel nitrate salt mixtures for thermal energy storage fluids. To achieve this, it focuses on experimentally determining the specific heat capacity of potassium nitrate (KNO_3), lithium nitrate ($LiNO_3$), and magnesium nitrate hexahydrate ($Mg(NO_3)_2{\cdot}6H_2O$), and assessing their potential as effective TES fluids. The outcomes from this study will contribute to cost effective and high performance by nitrate salt mixtures choice for

thermal energy storage fluid, hence improving suitability and efficiency of CSP techniques to support renewable energy system.

2.0 **Materials and Methods**

2.1 Materials and sample preparation

The nitrate salt mixture, consisting of potassium nitrate (KNO_3), lithium nitrate ($LiNO_3$), and magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$), was selected based on its thermal stability, favorable thermophysical properties, and availability for thermal energy storage fluid applications, as stated by Kwasi-Effah et al. [22]. The salts were procured from trustworthy suppliers at purity greater or equal to ninety – nine percent and used with no additional purification to retain their original industrial conditions.

2.1.1 Mixing Ratio Selection

Based on previous research by Kwasi-Effah et al. [22], the mix ratios were carefully chosen by percentage weight to optimize specific heat capacity and melting point. To balance these properties and expand the operating temperature range, the specific mixing ratios listed in Table 1 were selected. Other studies, including those by Ibrahim et al. [23] and Isaza-Ruiz et al. [24], have also implemented similar research methods. For this study, KNO_3, $LiNO_3$ and $Mg(NO_3)_2.6H_20$ variation selected were from 37% to 43%, 28% to 33% and 28% to 32% respectively proportion by weight to assess their impacts on the nitrate salt mixtures.

Table 1: Seven sample of ternary nitrate salt mixture ratios

S/N	Sample Name	KNO_3 (%wt)	$LiNO_3$ (%wt)	$Mg(NO_3)_2.6H_2O$ (%wt)
1	TSF1	42	29	29
2	TSF2	40	30	30
3	TSF3	38	31	31
4	TSF4	41	30	29
5	TSF5	39	31	30
6	TSF9	40	28	32
7	TSF10	40	31	29

2.2 Experimentations

The salts were weighed to a precision of ±0.001g using an analytical balance (Mettler Toledo XS205DU, 0.01 mg readability). This high precision balance allows accurately measuring sample sizes down to several milligrams. The balance was calibrated using standard 100 mg and 200 mg Mettler Toledo calibration weights prior to each usage session. The calibration was performed at room temperature to ensure weighing accuracy. The calibration process involves placing the calibration weights on the balance pan and initiating an internal calibration routine. The balance optimizes and linearizes the weighing parameters to minimize errors.

The calibration tolerance was within ±0.1 mg, enabling reliable weighing of the 7.2 mg salt samples to within ±0.001 g precision.

The balance was installed on a vibration-dampened table to minimize external disturbances during sample weighing. The surrounding environment was maintained at controlled room temperature. The 0.01 mg readability scale allows clear discrimination between the small gradations in sample compositions around the 40% KNO_3 region. This high weighing precision was essential for accurate characterization of property changes with salt composition.

The balance calibration, environmental control and precision capabilities helped ensure accuracy and repeatability for the sample preparation step during property characterization of the molten salt mixtures. The salts were thoroughly mixed using a high energy ball mill (SPEX 8000 M mixer/mill)

for 30 minutes to ensure homogeneity. This is essential for the salts to be uniformly distributed, which plays a critical role in the consistency of the thermal properties.
To remove any residual moisture from the salt mixture, a vacuum oven (Across International ADP-31) was used to dry the salt mixture for 24 hours at 120^{o}C and 50 mbar and then stored in airtight containers under a 99.999% pure Nitrogen atmosphere. This helps eliminate hydroscopic moisture that could affect the thermal properties and also prevent oxidation. Drying temperature of 120^{o}C and vacuum pressure of 50 mbar were selected to ensure complete removal of moisture without causing decomposition of the salts.
Specific heat capacities for various samples were measured by DSC (TA Instruments Q20) from 30°C to 400°C at 10°C/min, using ~10 mg samples in hermetic pans with a 25 mL/min N_2 purge. Sapphire standard was used. Melting temperature and latent heat was measured by DSC with 5°C/min heating rate under N_2 atmosphere.

2.3 Uncertainty Analysis

A comprehensive uncertainty analysis to quantity and account for possible source of uncertainty in the experiment measurements was carried out with an aim to ensuring the accuracy and reliability of the values of the thermal properties reported. Below are factors considered in the analysis:

i. **Weighing Uncertainty:** With proper calibration of the Mettler Toledo XS205DU with readability of 0.01mg before use, the weighing uncertainty is estimated to be $\pm$0.001g. This uncertainty affects the precision of the salt mixture composition.

ii. **Temperature Uncertainty:** The instrument for thermal diffusivity (LFA 447 NanoFlash) has a manufacturer's specified temperature accuracy of $\pm$3^{o}C. This temperature uncertainty influences the calculated thermal conductivity values.

iii. **Sample Preparation Uncertainty:** Sample inhomogeneity, incomplete mixing and residual moisture content are factors capable of inducing uncertainties in the sample preparation process. The thorough mixing and drying processes implemented in this work helps minimize these effects.

iv. **Instrument Calibration Uncertainty:** Standard reference sample Pyroceram 9606 was used to calibrate the LFA 447 instrument after every 10 measurements. Other measuring techniques included gas pycometry and differential scanning calorimetry also contributes to the instrument calibration uncertainties.

v. **Data Scatter Uncertainty:** Three measurements were taken at each temperature and the result averaged, in other to account for potential data scatter and improve accuracy. The standard deviation of these replicate measurements provides an estimate of the random uncertainty in the thermal properties.

The individual uncertainties from these sources were quantified and combined using appropriate statistical methods to determine the overall uncertainty in the reported thermal properties values. The combined standard uncertainty was calculated using root-sum-square method, and the expanded uncertainty was reported with a coverage factor of 2, providing a confidence level of approximately 95% as shown in Equation (1) [25].

$$U = k \times U_c \quad (1)$$

Where:

U is the expanded uncertainty; k is the average factor (k = 2 for 95% confidence level) and U_c is the combined standard uncertainty.

The combined standard uncertainty U_c is calculated by combining the individual standard uncertainties from various sources using the root-sum-square method:

$U_c = \sqrt{(u_1^2 + u_2^2 + u_3^2 + \cdots u_n^2)}$ (2)

Where $u_1^2, u_2^2 u_3^2 \ldots u_n^2$ are the standard uncertainties associated with different sources

2.4 Safety Considerations

Appropriate PPE including thermal gloves, face shield and lab coat were worn at all times during experimentations. Temperature probes were used to monitor the melt.

2.5 Suitability evaluation of Various Nitrate Salt Mixture for Thermal Energy Storage Fluid

The seven nitrate salt mixtures were subjected to suitability test based on specific heat capacity values to be obtained. The minimum specific heat capacity values suitable for TES fluid that can be used for CSP system is 0.5J/g-k as stated by Kouihen et al. [26]. The suitability was done by determine the different between the experimental values and minimum specific heat capacities as shown in Equation (3). The different in specific heat capacities (ΔCp) is expressed as:

$$\Delta Cp = Cp_e - Cp_m \quad (3)$$

where Cp_e and Cp_m are experimental and minimum specific heat capacities respectively.

When ΔCp outcomes is a positive value, the salt mixture specific heat capacity will be acceptable for thermal energy storage fluid.

3.0 Results and Discussion

3.1 Experimental Results

Measurements of specific heat capacities for seven ternary nitrate salt mixtures (TSF1, TSF2, TSF3, TSF4, TSF5, TSF9, TSF10) obtained from the DSC equipment are provided in form of specific heat capacity curves as shown Figures 1 to 7 respectively. The specific heat capacity curve is a graphical representation of how the temperature of a substance changes as heat energy is added or removed. The figures are variations of heat flow with temperature from the experimental procedure. The various specific heat capacities for the seven ternary nitrate salt mixtures, as determined by DSC, are also presented in Figures 1 to 7.

As shown in Figure 1 to Figure 7, the Cp values obtained ranged from around 0.5829 J/gK at 370K at the melting phase up 0.998J/gK at 370K across the different samples. TSF2 exhibited the highest Cp at the reference temperature of 370K with a value of 0.9987J/gK as seen in Figure 2, followed by TSF10 with a value of 0.858 J/gK as obtained in Figure 7. Additionally, the specific heat capacity of 0.6875 J/gK, 0.6040 J/gK, 0.5829 J/gK, 0.5845 J/gK and 0.5768 J/gK were obtained for TSF4, TSF5, TSF3, TSF 1 and TSF 9 respectively as presented in Figure 4, 5,3, 1 and 6. This highlights that small compositional variations can substantially impact C_p. The 40/30/30 TSF2 sample achieved the highest C_p at the reference temperature. Although similar KNO_3 content but slightly lower Li and Mg ratio, the TSF9 had far less C_p.

Additionally, the specific heat capacity (C_p) increases with increasing temperature, which is attributed to the enhanced ionic mobility and thermal vibration of the ions. As temperature rises, the ions gain kinetic energy and move more rapidly, leading to increased thermal vibration, which in turn enhances ionic conductivity, diffusion, and heat transfer.

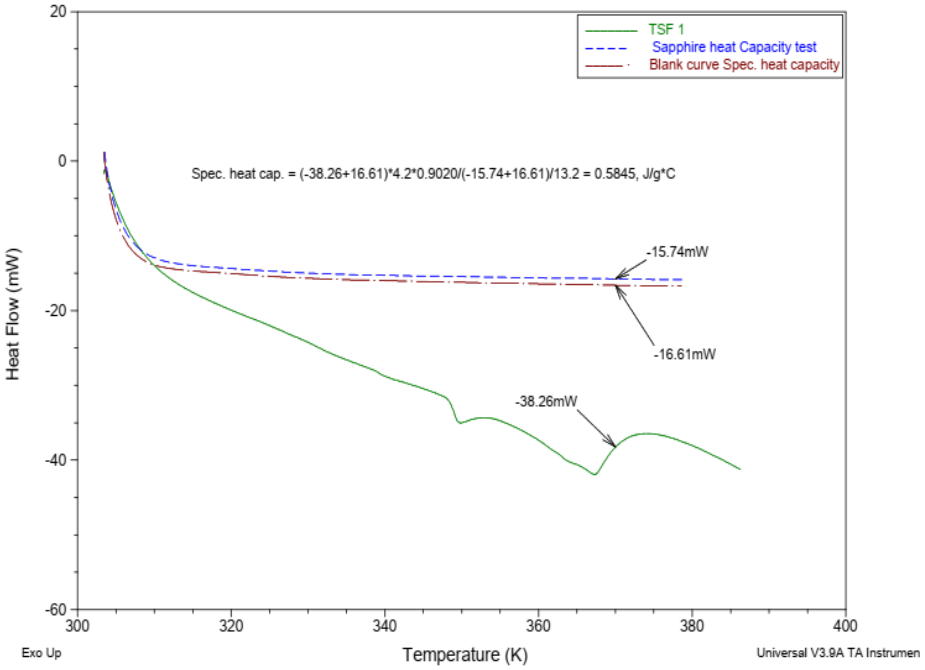

Figure 1: Specific heat capacity of TSF1

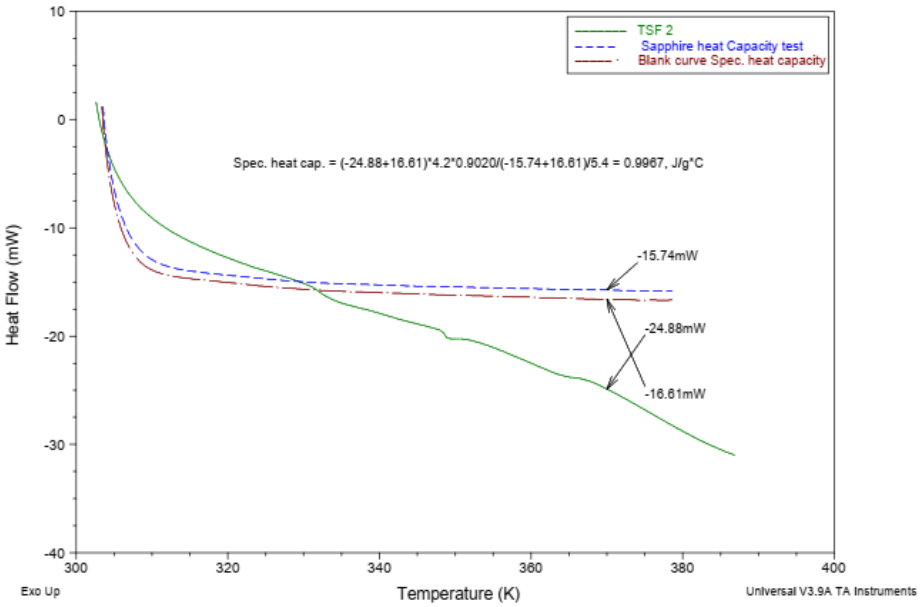

Figure 2: Specific heat capacity of TSF2

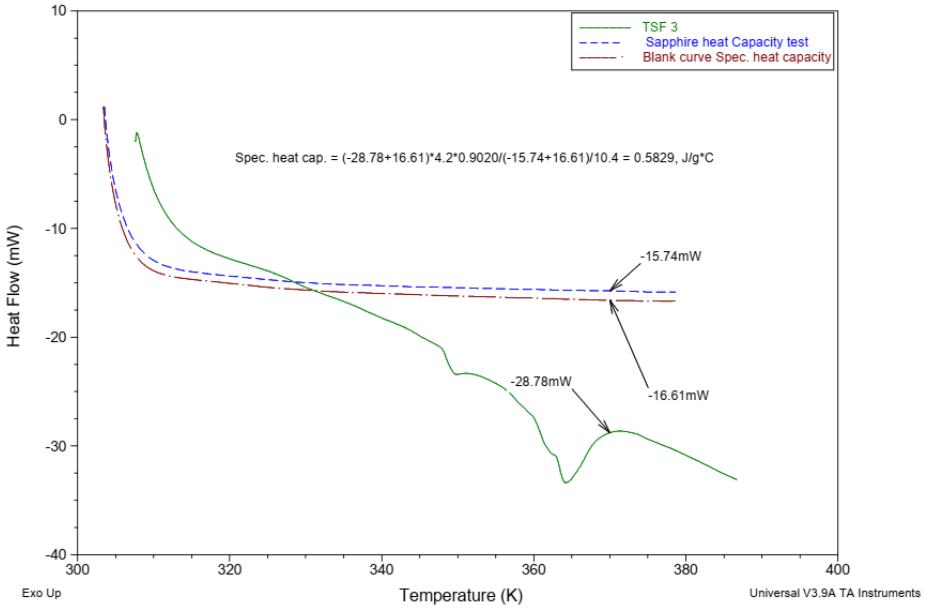

Figure 3. Specific heat capacity of TSF3

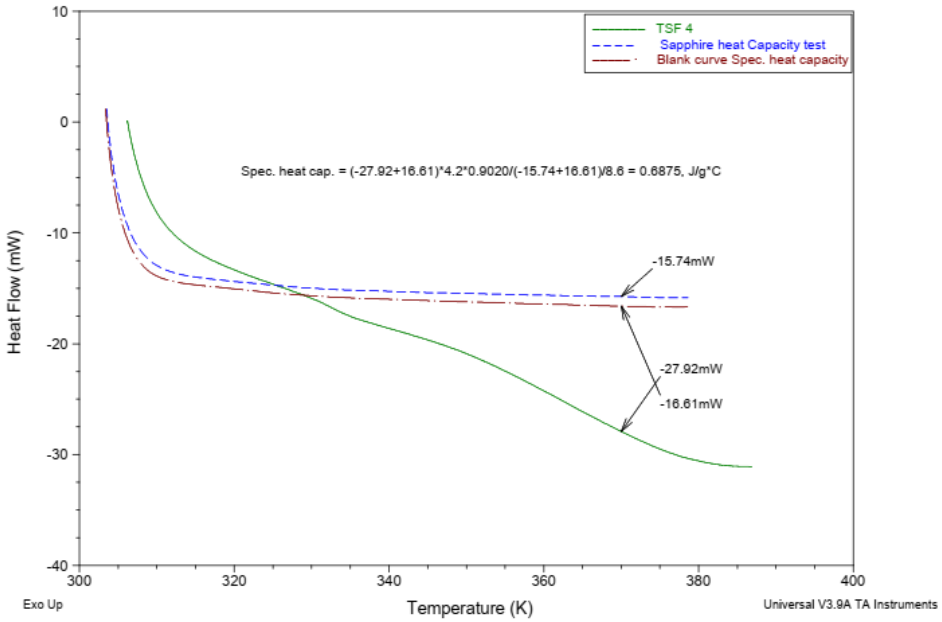

Figure 4: Specific heat capacity of TSF4

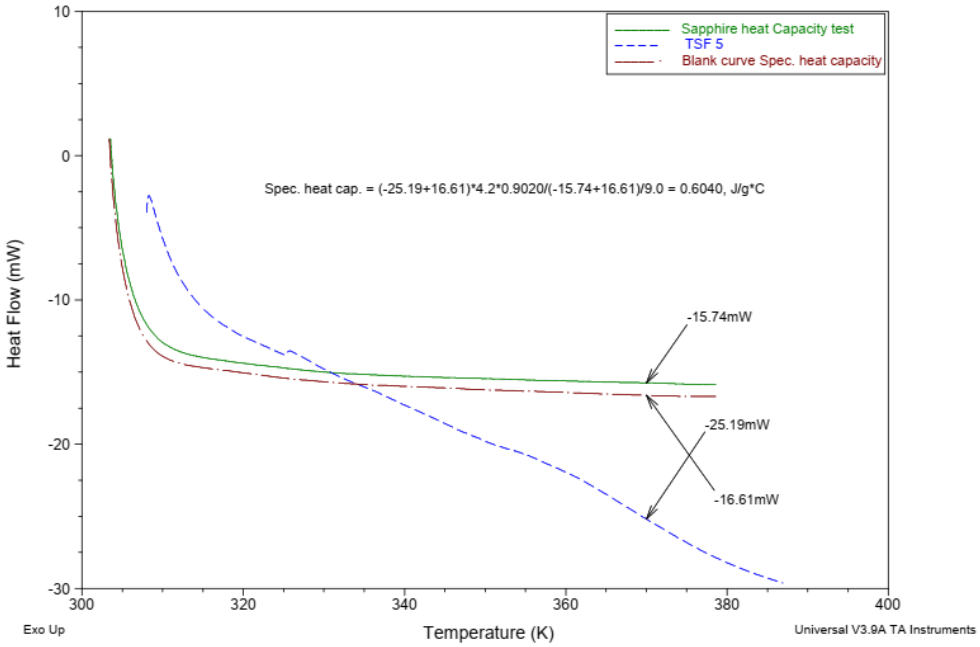

Figure 5: Specific heat capacity of TSF5

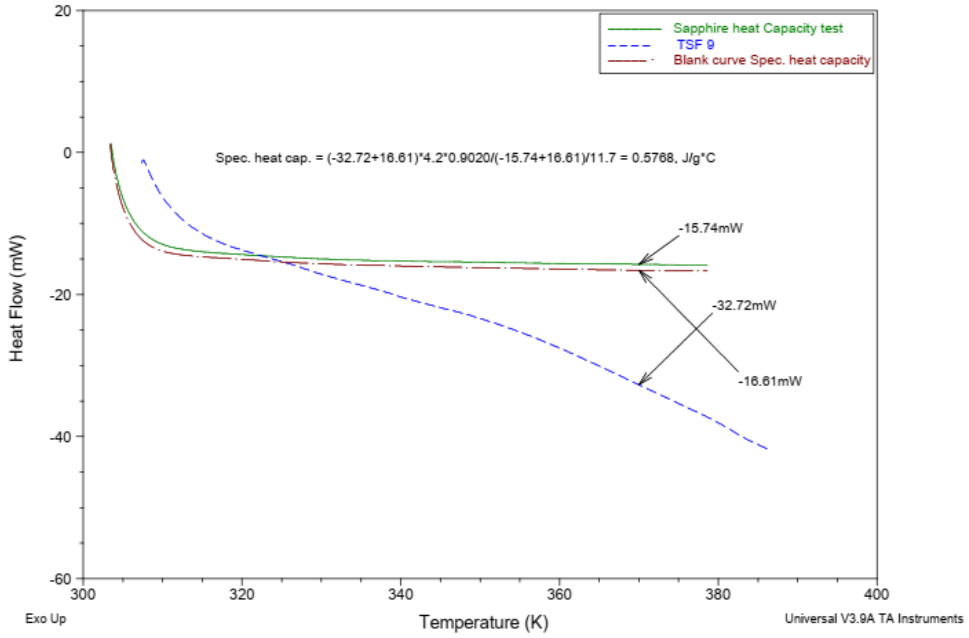

Figure 6: Specific heat capacity of TSF9

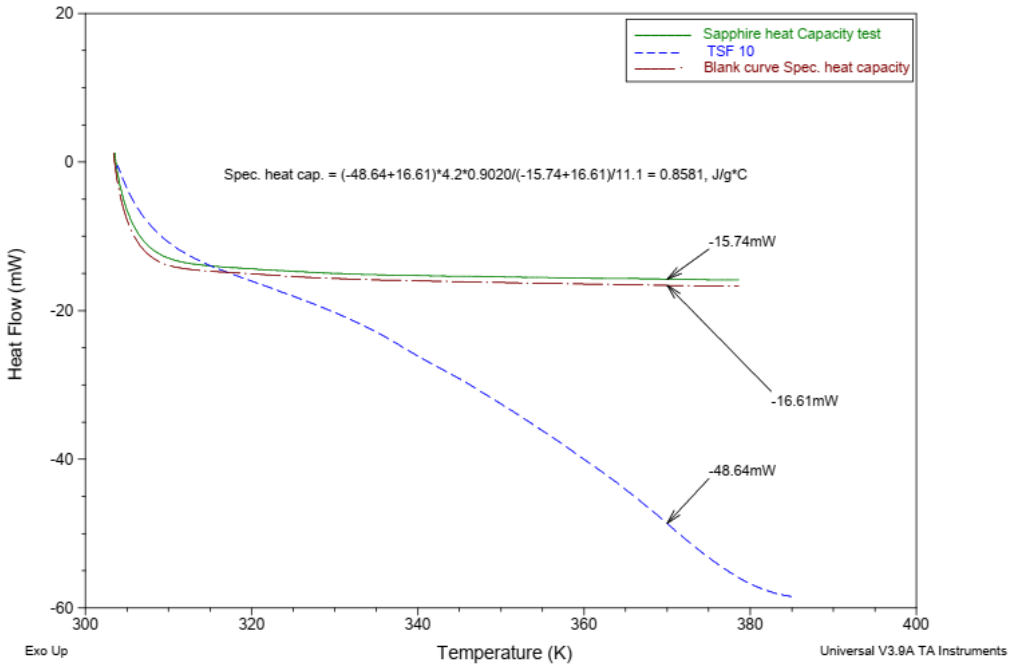

Figure 7: Specific heat capacity of TSF10

When the ions become more mobile, they can absorb and store energy more efficiently, distribute heat more evenly throughout the substance, and facilitate thermal relaxation processes. This enables the substance to absorb, store, and transfer heat energy more effectively, resulting in an increase in specific heat capacity. Furthermore, this behavior demonstrates that all seven nitrate mixtures of potassium, lithium, and magnesium salts developed exhibit good heat transfer abilities.

3.2 Suitability of Various Nitrate Salt Mixture for Thermal Energy Storage

The suitability results for the different Nitrate salts mixture obtained using Equation (3) are presented in Table 2.

Table 2: Various Nitrate Salt Mixtures Suitability Results

S/N	Sample Name	cp_e (J/gk)	Cp_m (J/gk)	Δcp (J/gk)
1	TSF1	0.5845	0.500	0.085
2	TSF2	0.9987	0.500	0.499
3	TSF3	0.5829	0.500	0.083
4	TSF4	0.6875	0.500	0.188
5	TSF5	0.604	0.500	0.104
6	TSF9	0.5768	0.500	0.077
7	TSF10	0.8581	0.500	0.358

As shown in Table 2, the difference between the experimental and minimum specific heat capacities obtained are all positive values. This indicates that all the seven mixtures of the nitrate salt developed can be used for thermal energy storage fluid in CSP systems based specific heat capacity values obtained. Thus, measurements of specific heat capacities for seven ternary nitrate salt mixtures (TSF1, TSF2, TSF3, TSF4, TSF5, TSF9, TSF10) provided valuable insights into how variations in composition for thermal energy storage in concentrated solar power plants. The results showed significant variations in specific heat capacities among the salt mixtures, emphasizing the need to optimize composition for desired performance.

4.0 Conclusion and Recommendation

This study focused on determining the specific heat capacity of novel ternary nitrate molten salt mixtures, based on phase change materials, for thermal energy storage in concentrated solar power systems. Seven developed thermal storage fluid mixtures were considered, using molten inorganic nitrate salts. The ternary nitrate mixture composed of 40% KNO_3, 30% $LiNO_3$, and 30% $Mg(NO_3)_2{\cdot}6H_3O$ exhibited an excellent specific heat capacity of 0.9987 J/g·K, and all mixtures showed favorable heat capacities exceeding 0.5 J/g·K. Thus, all the investigated ternary nitrate mixtures demonstrated their suitability for thermal energy storage (TES) utilization in CSP system applications. The results revealed significant variations in specific heat capacities among the salt mixtures, emphasizing the need to optimize composition for desired performance. To reduce costs and reliance on extensive experimental testing of ternary nitrate salt mixtures, this study suggests that a predictive model should be developed to estimate the specific heat capacity of various nitrate salt mixtures across a wide range of compositions.

Conflict of Interest

The authors declare no conflicts of interest, financial or otherwise, that could potentially influence or bias the research work presented in this paper.

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