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Characterization and Heat Transfer Performance of Quarternary Nitrate Based Molten Salts

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ABSTRACT

The use of molten salts as heat transfer has become the preferable composition. It has excellent properties such as low melting points, high heat capacity, and a wide range of temperatures. This paper focuses on the characterization and heat transfer performance of quaternary nitrate based on molten salts whose composition has been reported by previous researchers. The quaternary molten salts ($LiNO_3$, $NaNO_3$, KNO_3, $Ca(NO_3)_2$) were characterized to determine the melting point and heat capacity using Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC), respectively. The heat transfer performance was then tested on the test rig of the designated system to determine the heat transfer properties of molten salts. The composition of 10 wt% $LiNO_3$, 10 wt% $NaNO_3$, 40 wt% KNO_3, 40 wt% $Ca(NO_3)_2$ has a low melting point of 97.7°C, high heat capacity which was 0.46J/g°C, and maximum thermal stability temperature was 439.04°C. The heat transfer performance test showed that this sample can be used as heat transfer fluid and can store energy in the system. In conclusion, quaternary nitrate-based molten salt is a promising candidate for heat transfer and energy storage in heat recovery applications.

1. Introduction

There has been an increasing interest in utilizing low-temperature waste in recent years. The low-temperature waste heat from solar thermal, geothermal, biomass, industrial and automobile, are among the potentially promising energy resources capable of meeting today's world energy demand [1]. This kind of heat needs to be extracted and stored in the same process or different to produce useful energy through the energy recovery system. Therefore, the capability of the heat transfer cycle in the system needs to be much concern. Heat transfer fluid is an important element to be considered to enhance the heat transfer operation in the energy recovery system. Fluids are expected to have high thermal conductivity, high volumetric heat capacity, and low viscosity for heat transfer

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performance. They also need to be environmentally benign, non-corrosive, safe, and cost-effective [2].

In this sense, molten salts are best positioned to be used as heat transfer fluids (HTF) and thermal energy storage materials (TES) [3]. Molten salts are currently being investigated as good heat transfer and thermal storage materials in energy storage applications, i.e., concentrated solar power (CSP) technology [3]. Molten salt is used because it is a liquid at atmospheric pressure, non-flammable, low cost, and efficient store energy. Besides that, molten salts also offer unique advantages such as low vapor pressure, safe operation, minimal environmental footprint, and moderate cost. [4]. Among the mixture of molten salts, the eutectic mixture of salts exhibits the lowest melting point from any of the same mixture with the same components. This kind of molten salt possesses excellent transport and thermal properties. Molten salts can be categorized into four mixture types that previous researchers have studied. There are include primary salt, binary salt [5], ternary [6], and quaternary salt [7]. Among these salts, a group of quaternary molten salt compositions has been identified that display a much lower melting point useful as heat transfer fluid and energy storage for heat recovery systems. Therefore, the characterization of the various molten salt mixture should be reviewed to obtain a lower freezing point with high thermal stability than the molten salt mixture used previously as a heat transfer application.

In nitrate salt mixtures, eutectic points exist, where at specific chemical composition, the system solidifies at a low temperature at any other composition. Two components of the liquid mixture are in equilibrium, and each component is crystal. However, if the temperature is lower than the eutectic temperature, each component will crystallize out of the mixture. Using salt with a near eutectic composition will have a lower melting point, high thermal stability, and high heat capacity than using pure salt. To understand the mechanisms involved in improving the functioning of the nitrate salt mixture, it is important to identify the thermal properties of pure components and binary mixtures. For binary mixture, KNO_3-$NaNO_3$ is commercially used for some applications such as transfer fluid which acts as a heat storage medium in the molten salt TES tank. Single salt of $NaNO_3$ melts at 307ºC, and KNO_3 melts at 337ºC. The proportion of solidus and liquidus generally agreed that the composition of KNO_3 (54 wt%)-$NaNO_3$ (46 wt%) fortunately there has always been agreement that the minimum melting point was close to 220°C. The binary mixture of KNO_3-$NaNO_3$ is also stable up to 500°C and has little weight change of the melt, although, over this temperature, there were some evolved NOx gases.

In a paper written by Fernández *et al.,* [8], adding or replacing lithium nitrate or LiNO3 with NaNO3 can improve the performance of molten salt extending the temperature work range regarding a low melting point higher thermal stability. Eutectic behavior and more drastic melting point reduction occur with a more complex salt mixture such as ternary. Cordaro and Rubin [6] proposed extending the upper working temperature by adding the other salt or controlling the nitrate to nitrite ratio to reduce the binary mixture's melting point. For example, molten mixture is usually defined as KNO_3 (53 wt%)-$NaNO_2$ (40wt%)-$NaNO_3$ (7 wt%), or KNO_3 (44 mol%) - $NaNO_2$ (49 mol%)-$NaNO_3$ (7 mol%). Consequently, new heat transfer fluids with various additions of $Ca(NO_3)_2$ and $LiNO_3$, replacing the binary salt currently used. Therefore, reducing the melting point or improving the characteristic of the salt mixture in a binary mixture [9] has highlighted that adding or replacing $LiNO_3$ in the ternary system in KNO_3-$NaNO_2$-$NaNO_3$ is a suitable characteristic in improving the range of thermal stability of salt. However, the main problem associated with this additive is price.

The one addition of molten salt that can reduce the melting point and improve the binary salt mixture is $Ca(NO_3)_2$. $Ca(NO_3)_2$ is one of the additives that low cost and can reduce the melting point of alkaline nitrates. It demonstrates the additive's enormous potential to be included in new formulations of molten salts for energy storage. It makes it a primary candidate to substitute the binary solar salts—the minimum melting temperature of this system found with value 160ºC. Wang *et al.,* [10] proposed a new quaternary mixture with a composition of 10 wt% $LiNO_3$+20 wt% $NaNO_3$+60 wt% KNO_3+10 wt% $Ca(NO_3)_2$ exhibits better physical and chemical properties than the binary solar salt currently used. Besides that, Bradshaw *et al.,* [9] studied that is known to disclose anhydrous compositions mixture belonging to the quaternary $LiNO_3$-$NaNO_3$-KNO_3-$Ca(NO_3)_2$ system having a melting temperature below or closely 95°C and high thermal stability up to the temperature of 500 °C. Another research by Kearney and Mahoney [11] investigated this quaternary mixture of heat capacity. The analysis was performed using the modulated DSC and revealed the heat capacity is 1.518 J/g ºC and which improves the heat capacity binary mixture currently used. However, the study about the relationship between the molten salt characteristics (melting point, heat capacity) and heat transfer performance was still limited. In this study, the characterization and heat transfer performance test of quarternary nitrate-based molten salts was carried out to determine their relationship and proposed heat transfer data for the selected composition of quarternary nitrate-based molten salts from previous literature.

2. Methodology

2.1 Materials and Preparation

Four primary nitrate-based molten salts have been used to study the characteristics and heat transfer performance. The primary molten salts included Lithium Nitrate (LiNO3, 99.8%, HmbG® Chemicals, Grade PRS), Sodium Nitrate ($NaNO_3$, 99.8%, ChemAR®, Grade A.R), Potassium Nitrate (KNO3, 99.8%, PC Laboratory Reagent, Grade A.R) and Calcium Nitrate (Ca(NO3)2, 99.8%, Bendosen, Grade A.R). The composition of quarternary nitrate-based molten salts has been chosen from the previous researcher [9]. These compositions are selected based on the low range of meting point (below 100°C) that has been conducted in their experiment. The details of the composition as shown in Table 1.

Table 1
Composition of samples in wt%

Sample No	$LiNO_3$ (wt%)	$NaNO_3$ (wt%)	KNO_3 (wt%)	$Ca(NO_3)_2$ (wt%)	Reference
1	17.6	13.7	44.1	24.5	[12]
2	10	10	40	40	
3	10	10	60	20	[13]
4	20	10	50	20	

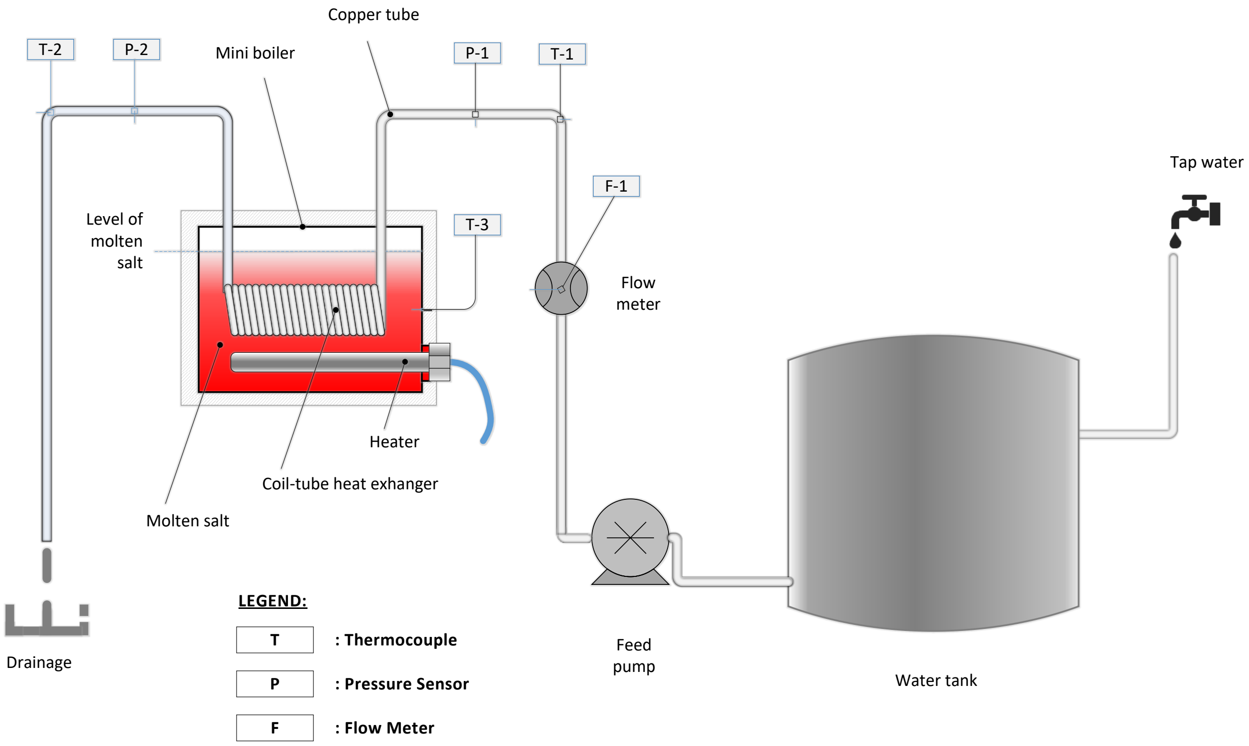
The primary molten nitrate salts have been weighed and must undergo a drying process inside the oven or furnace at 100°C for 24hours to evacuate any bounded water or wetness. The quaternary molten nitrate salt mixture was then heated inside a furnace, and the details of the heating profile and preparation of the sample are given in an earlier paper [14].

2.2 Characterization

After the quaternary molten salts have been prepared, the several testings were carried out to determine the thermal properties of each sample mixture. Differential scanning calorimeter (DSC) testing was carried out to determine the heat capacity with its melting point. For the results of the liquidus temperature and maximum thermal stability temperature, the thermogravimetric analysis (TGA) has been conducted.

2.3 Heat Transfer Performance Test

The heat transfer performance test for quaternary molten nitrate salt mixtures was analyzed using a designated system to determine the heat transfer rate in kilowatt (kW). The system consists of several components that are installed together: the water pump, flow meter, boiler, and heater. The control panel attached to the system with the heater, temperature reading, and flow rate controller were used for data collection. In the system, tap water is used as a working fluid (tap water), whereas quaternary nitrate-based molten salt is a heat transfer fluid that flows along the pipeline. The heat transfer performance test began as the quaternary molten nitrate salt mixtures were filled inside the boiler until they reached the point of the molten salts level. The Figure 1 shows the schematic diagram flow of the heat transfer in the system.



(a)

(b)

Fig. 1. Molten Salt Heat Transfer Test System (a) schematic diagram; (b) photo of configuration

According to the schematic diagram, the working fluid flow started at the source of tap water. Then, the flow continued by entering the boiler through a copper coil and flowing out from the boiler into the drainage. The working fluid acts as a heat carrier from the quaternary molten salts inside the boiler. The flow rate data with different ranges of the working fluid, outlet (T2), and inlet (T1) temperature were recorded by every five minutes interval for 60 minutes. The value of heat transfer rate, Q, was calculated using the following Eq. (1).

$$Q = \dot{m}\ (h2 - h1) \tag{1}$$

where, mass flow rate, $\dot{m}$, enthalpy at outlet temperature in kJ/kg unit, h2 and enthalpy at inlet temperature in kJ/kg unit, h1 respectively

The enthalpy difference (h2-h1) needs to be determined to get the heat transfer rate according to the equation. The enthalpy value was collected through REFROP software according to the value of the outlet (T2) and inlet (T1) temperature of the working fluid. In this study, three different range of flow rate has been applied to determine the heat transfer rate value. This flow rate range was determined by the minimum and maximum value adjusted using the ball valve. The range of flow rate is as shown in Table 2.

Table 2
Range of flow rate

Range	Value (ℓ/min)
Low	0.1<x<0.2
Medium	0.2<x<0.4
High	0.4<x<0.7

Two conditions were conducted during the test. The "Heater On" condition, known as a steady-state condition, was done to determine the heat transfer rate of quaternary molten salts according to the flow rate range. The "Heater off" condition was conducted to determine the ability of quaternary nitrate-based molten salts to store heat energy inside the system against time.

During the steady-state condition (Heater on), the first step for this testing was to set the heater's temperature at 100oC. After that, by setting the flow rate range into the low, medium, and high range, the value of outlet, T2, and inlet, T1 temperature was recorded. At the same time, the actual heater temperature was also recorded according to the flow rate range to confirm the temperature was precisely stable to the temperature of the quaternary molten nitrate salts mixture.

The testing was repeated by setting the temperature of the heater for 125°C, 150°C, 175°C, and 200°C. The value of enthalpy differences and the heat transfer rate in kW were then calculated. The "Heater off" condition data was collected starting at a temperature setting of 200°C after the steady-state condition was completed. The flow rate range was set at the low working fluid flow rate range, which was between (0.1 < flow rate < 0.2) liter/minute. The result of quaternary molten nitrate salts temperature inside the boiler (Tm), the value of outlet (T2), and inlet (T1) temperature was recorded every five minutes for one hour. The value of enthalpy differences and the value of the heat transfer rate in kW were then calculated.

3. Results

3.1 Melting Point and Heat Capacity

The DSC curve of various composition has been reported in previous research [12]. The details of DSC curve for selected samples are shown in Figure 2.

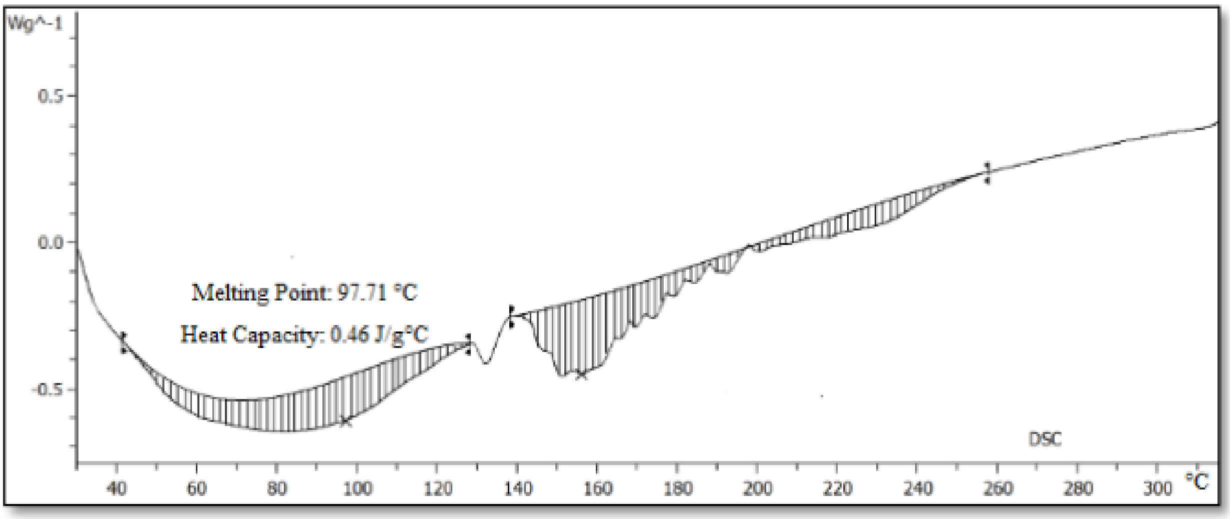


Fig. 2. DSC curve of sample 2 (10 wt% $LiNO_3$, 10 wt% $NaNO_3$, 40 wt% KNO_3, 40 wt% $Ca(NO_3)_2$

Based on the result from the DSC curve, two peaks have been detected throughout the testing. The first peak of the DSC curve refers to the value of melting point and heat capacity of the samples. The value of melting point for all samples were below 100°C which is same as reported by Bradshaw [15], and Ren *et al.,* [13]. Table 3 shows the experimental data and literature data.

Table 3
Comparison Experimental data and literature data of melting point for quaternary nitrate-based molten salts

Sample No.	Literature (T_m, °C)	Experiment (T_m, °C)	ΔT (°C)	Reference
1	<95	96.74	1.74	[12]
2	62.8	97.71	34.91	
3	80.4	77.83	-2.57	[13]
4	85.4	81.12	-4.28	

As Table 3, there are quite differences between experimental and literature data. According to Bernagozzi *et al.,* [16], the difference in the value is due to three reasons. Due to the precision composition required for the primary salts during weighing before mixing, the complexity is associated with an increase of individual mixture components and the purity of the primary salts. However, all the values could be acceptable as the low melting molten salts, which the melting point was below 100°C.

A low melting point with high heat capacity properties is needed for good heat transfer fluid in the heat recovery system. The conventional heat transfer fluid, which was solar salt (binary mixture) and Hitec salt (ternary mixture), has been used commercially with a melting point of 220°C. The significant problem that might be occurred by using high melting point molten salts as heat transfer fluid is that it can freeze at high temperatures [17]. Therefore, a low melting point of molten salt was desired to prevent the problem, and quarternary nitrate-based molten salts could be a potential candidate for heat transfer fluid.

Figure 3 shows the heat capacity value of quaternary nitrate-based molten salt. As seen from the Figure 3, the highest value of heat capacity obtained from Sample 2 (10 wt% $LiNO_3$, 10 wt% $NaNO_3$, 40 wt% KNO_3, 40 wt% $Ca(NO_3)_2$) whereas the lowest value is from Sample 3 (20 wt% $LiNO_3$, 10 wt% $NaNO_3$, 60 wt% KNO_3, 20 wt% $Ca(NO_3)_2$. According to Pfleger *et al.,* [18], a mixture with a low melting point and high heat capacity have an excellent characteristic that needs to be considered in a salt system as a storage medium and heat transfer fluid. Nunes *et al.,* [19] also mentioned that as the value of heat capacity of the system is higher, better thermal energy storage capacities.

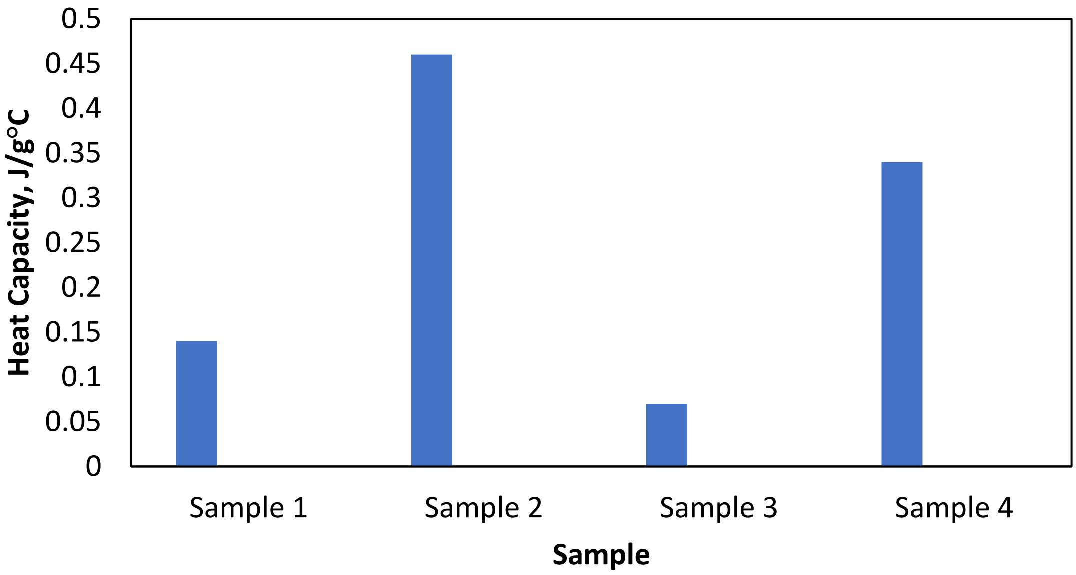


Fig. 3. Heat capacity for quaternary nitrate based molten salts

3.2 Thermal Stability

Thermogravimetric analysis (TGA) experiments provide a first approach to understanding quarternary nitrate-based molten salts [5]. The thermal stability property determined the upper limit of the working temperature of quaternary molten salts. Figure 4 shows the TGA curve for Sample 2. Based on the figure, the sample started to degrade at 36.03°C, and the highest weight loss was 4.04mg compared to the other samples. The large weight loss below 200°C is due to evaporation of absorbed water [20]. Meanwhile, the maximum thermal stability temperature of Sample 2 is the highest. As compared to other samples (Table 4), the value of maximum thermal stability increases from Sample 2 > Sample 4 > Sample 3 > Sample 1. It is related to the value of heat capacity. It is mentioned that the higher the heat capacity, the greater the thermal stability that satisfied the application in energy storage [10].

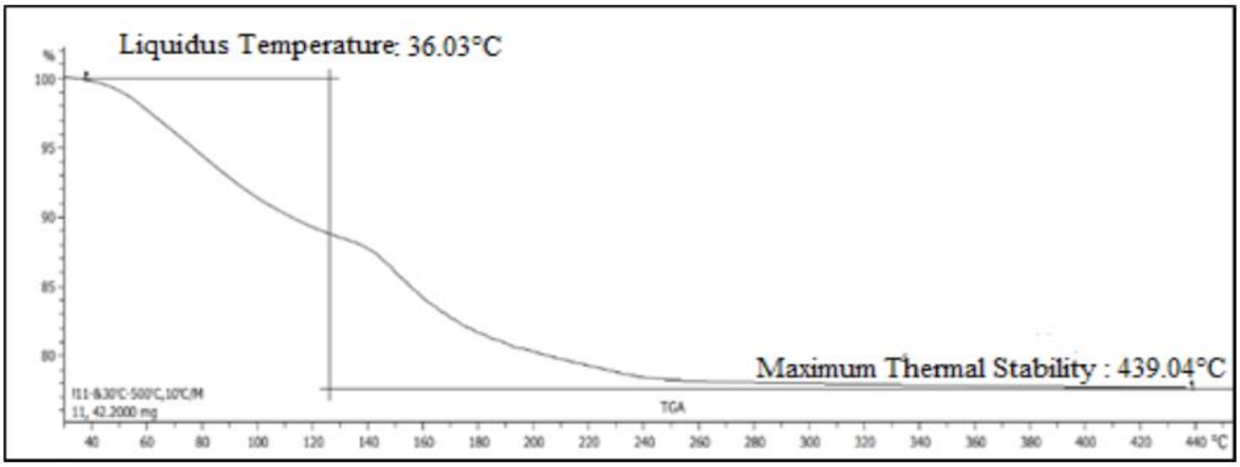


Fig. 4. TGA curve of Sample 2 10 wt% $LiNO_3$, 10 wt% $NaNO_3$, 40 wt% KNO_3, 40 wt% $Ca(NO_3)_2$

Table 4
The value of maximum thermal stability temperature of molten salts

Sample	Maximum Thermal Stability Temperature (°C)
1	319.87
2	439.04
3	377.83
4	396.77

3.3 Heat Transfer Performance Test

As mentioned before, heat transfer performance test has been conducted in two conditions: ‘Heater On’ condition (steady state) and ‘Heater Off’ condition. Setting temperature that has been set during the experiment were 100°C, 125°C, 150°C, 175°C and 200°C. This heat transfer performance analysis is necessary to study the ability of quaternary nitrate based molten salts in transferring and storing heat energy.

As seen in Figure 5, the heat transfer rate for all samples increased with increasing setting heater temperature at all flow rate ranges. The purpose of using different flow rates was to observe the heat transfer rate from HTF to storage medium [21]. The graph trend for all samples shows the same trend of heat transfer rate that has been reported by previous researchers that used other types of heat transfer fluid [22]. Increasing the heat source and mass flow rate will increase the heat transfer rate of molten salts. For the “heater off” experiment, water was used as working fluids for drawing the heat from the storage tank. The storage temperature started at 200°C. The period of the experiment was until 60 minutes after the heater controller had been shut down. The flow rate of working fluid is fixed to the low range. Even though the mass flow rate is fixed, the heat transfer rate will increase as mentioned in the “Heater On” condition.

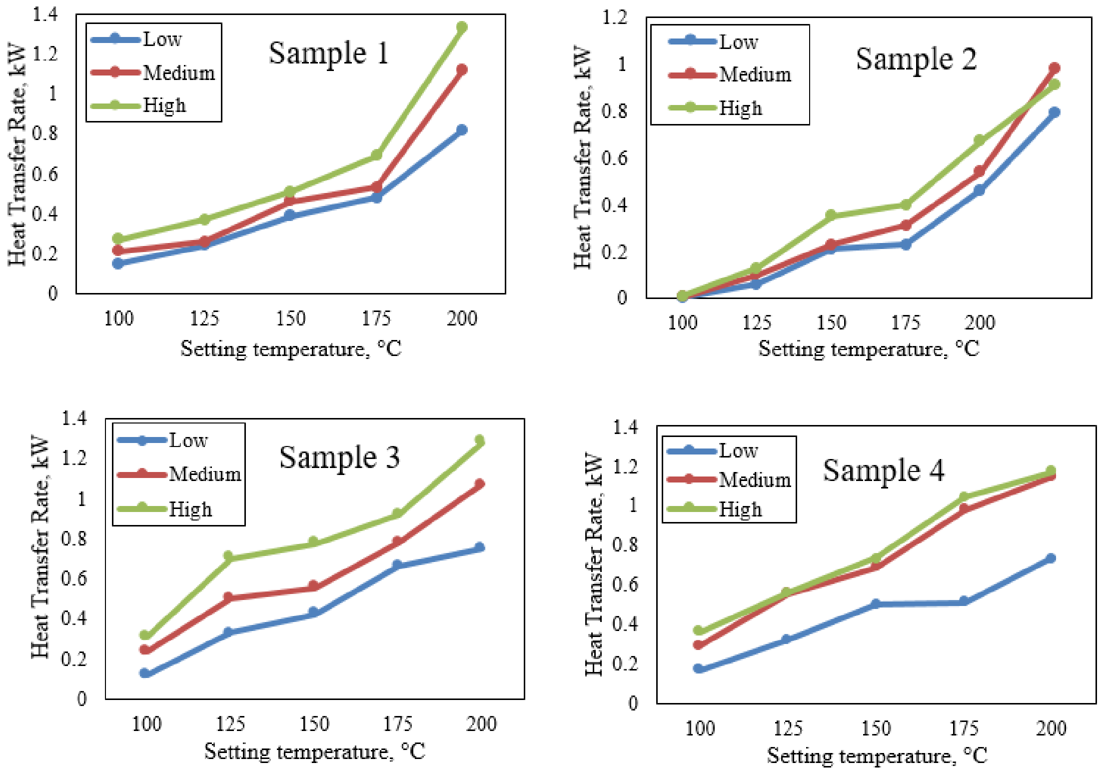


Fig. 5. Heat transfer rate for all samples at 100°C, 125°C, 150°C, 175°C and 200°C during "Heater On" condition

The reaction involved in this testing was an exothermic reaction where the heat was released from the quaternary molten nitrate salt mixture to the surrounding. As seen in Figure 6, during the first 35 minutes, there is a noticeable change in heat transfer rate since it appears that the heat loss to the surrounding was reasonably constant. However, after 35 minutes, the heat transfer rate decreased until it reached the equilibrium temperature, which was zero Celcius (0°C). From Figure 6, only Sample 1 and Sample 4 have reached the equilibrium temperature between inlet and outlet temperature after 60 minutes of the experiment. As mentioned earlier, Sample 1 and Sample 4 have low heat capacity values, 0.14 J/goC and 0.07 J/goC. Therefore, it can be concluded that low heat capacity could decrease the ability of the quaternary molten nitrate salt mixture to store heat.

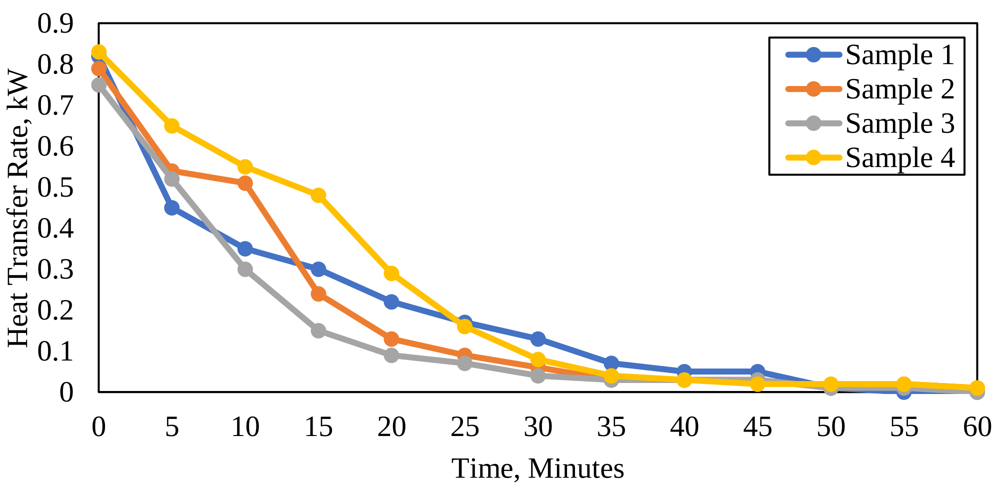


Fig. 6. Heat transfer rate of all samples during 'Heater Off' condition

Figure 7 show the temperature of quaternary nitrate based molten salts as function of time for all samples during 60 minutes of testing. From the figure, temperature of Sample 2 was decreasing slowly as compared with other samples. This result is clearly seen by refer the temperature difference between initial temperature at the beginning of experiment and the final temperature at the end of the experiment.

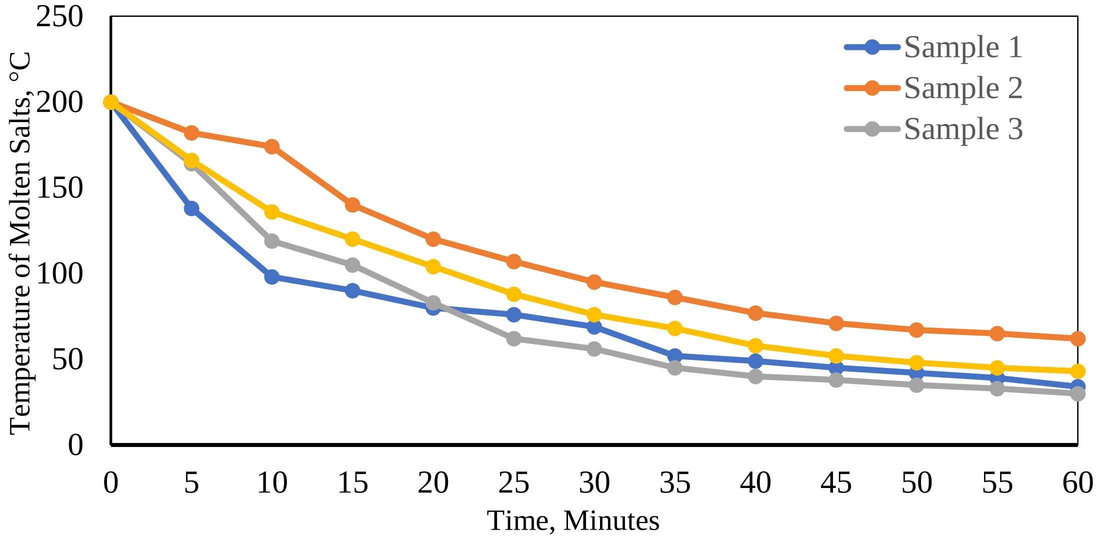


Fig. 7. Temperature of molten salts during 'Heater Off' condition

Table 5 demonstrate the value of heat loss from quaternary nitrate-based molten salts to the surrounding. Sample 2 shows the lowest temperature difference related to the high heat capacity, which was 0.46J/g°C. The low-temperature difference indicates that the ability of molten salts to store heat energy was increased. According to Wang *et al.,* [10], the value of heat capacity increased, the thermal stability of quarternary molten salts was higher in the application of heat energy storage [21]. The heat transfer rate of molten salts is slower due to their heat capacity value.

Table 5
Temperature Difference of quaternary nitrate based molten salt during "Heater off" condition

Sample	Initial Temperature (°C)	Final Temperature (°C)	Temperature Difference (°C)
1	200	34	166
2	200	62	138
3	200	30	170
4	200	43	157

4. Conclusions

The quaternary nitrate-based molten salts mixture has been prepared to analyze the TGA, DSC, and heat transfer performance test. The significant findings of this study have been highlighted as follows. Sample 2 with a composition of 10.00 wt% $LiNO_3$ + 10.00 wt% $NaNO_3$ + 40.00wt% KNO_3 + 40.00 wt% $Ca(NO_3)_2$ has a low melting point was 97.7°C, high heat capacity which was 0.46J/g°C, and maximum thermal stability temperature was 439.04°C. The determination of the heat transfer rate for quaternary molten nitrate salts has been done using the designated system with two conditions which were "Heater on" and "Heater off." During the "Heater on" condition, it is shown that as the flow rate increased, the heat transfer rate has also increased. For the "Heater off" condition, the temperature difference of quaternary nitrate-based molten salt temperature from the beginning to the end of the test has been determined. The temperature difference indicates the ability of quaternary molten nitrate salts to store heat energy. As the temperature difference was higher, the ability of quaternary molten nitrate salts to store heat energy has increased. From the “Heater off” analysis, Sample 2 shows good ability in storing heat energy since it loses the least heat compared to other selected samples. Therefore, it is proven that quaternary nitrate-based molten salts are promising candidates as heat transfer fluid in the heat recovery system.

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