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SCREENING, THERMAL CYCLING AND CONTAINER COMPATIBILITY ANALYSIS OF INORGANIC PHASE CHANGE MATERIALS (PCMS) FOR HIGH TEMPERATURE (200 TO 400^{O}C) THERMAL ENERGY STORAGE IN SOLAR COOKERS

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ABSTRACT

Solar energy offers clean and sustainable pathway for cooking; however, the inability of conventional solar cookers to operate beyond daylight hours severely limits their practical deployment. Integrating thermal energy storage (TES) based on phase change materials (PCMs) can overcome this limitation by storing excess solar heat and releasing it during periods without solar radiation. The identification of safe, efficient, and economically viable PCMs is therefore critical for advancing solar cooking technologies. This study systematically evaluates inorganic PCMs for latent heat TES in solar cookers. Twenty-nine candidate materials were pre-screened using health hazard classification (NFPA classes 0–2), latent heat capacity (>100 J g^{-1}), and economic considerations. Four materials—$NaNO_3$, KNO_3, $NaNO_3/KNO_3$ (solar salt), and NaCl/KCl—met the initial criteria. Thermo physical assessment excluded NaCl/KCl due to its high melting temperature (≈630 °C), unsuitable for cooking applications. The remaining PCMs were characterized using Fourier Transform Infrared Spectroscopy (FTIR) and Differential Scanning Calorimetry (DSC) to determine chemical stability and phase transition properties. Thermal reliability and container compatibility were evaluated through 100 heating–cooling cycles of solar salt in contact with stainless steel and aluminum. Solar salt retained 88.3% of its initial latent heat storage capacity and structural integrity, demonstrating strong thermal stability. Compatibility tests indicate that stainless steel is suitable as a storage container, whereas aluminum exhibits adverse interactions with the PCM. Overall, $NaNO_3/KNO_3$ (solar salt) emerges as a safe, cost-effective, and thermally stable PCM for long-term TES in solar cookers, enabling operation beyond daylight hours and enhancing practicality of solar cooking systems.

Keywords: Aluminum, Stainless Steel, Health Hazard, Chemical Stability, Cycling Stability

INTRODUCTION

Globally, there exists an imperative for achieving a balance between energy supply & demand, environmental conservation, and economic advancement due to increasing energy consumption, diminishing fossil fuel reserves, and ecological deterioration instigated by the combustion of fossil fuels. Consequently, countries across the globe are actively pursuing alternative renewable energy sources, including solar, wind, hydro, tidal energy and geothermal energy, among others (Mohamed *et al*., 2017). As indicated by the findings of Mofijur *et al.* (2019), there was a 1.5% growth in global primary energy consumption in 2018 when compared with the consumption levels noted in 2017. According to data from British Petroleum from 2011, 41% of the energy produced was used in buildings and residential areas. The home sector experiences high energy consumption from cooking. About 30% of the total energy consumed in many developing nations goes toward cooking. Identifying the most effective and cost-efficient cooking systems that utilize renewable energy sources is crucial in light of the existing energy limitations and the necessity to mitigate the extensive deforestation occurring in those nations. For an extended period, solar cookers have been recognized as a viable solution to the global challenge of diminishing fuel supplies and other ecological concerns associated with the reliance on fossil fuels for cooking purposes (Omara *et al.*, 2020). Researchers around the world have produced many classes of solar cookers including Direct and Indirect (or box type) solar cookers mainly for outdoor applications, as well as Advanced solar cooker types more suitable for the indoors. However, even the modern solar cooker, which are more expensive cannot run at night. Consequently, there exists a necessity for an energy storage system to accumulate thermal energy for utilization during the evening hours (Faidah, 2009). A review of existing literature (Haillot *et al*., 2011; Usman *et al.*, 2023) shows that thermal energy can be conserved through three principal methodologies: latent heat, sensible heat, and reversible thermochemical reactions. As articulated by Patel *et al*. (2017), Latent Heat Storage (LHS) is regarded as one of the most effective techniques for energy conservation and subsequent reutilization, employing phase change materials (PCMs). PCMs are substances possessing the capability to absorb (store) and release thermal energy. When the temperature drops, it releases the latent heat it has absorbed from the environment (Patel *et al.*, 2017). When PCMs melt, they endothermically absorb heat. When the PCM freezes, an exothermic process occurs, releasing the heat when required.

To lower the operational and maintenance cost of solar cookers, it's imperative to consider factors such as materials cost, availability, and health hazard when selecting PCMs for a desired application. Maldonado *et al*. (2018) carried out a comprehensive examination of materials derived from existing literature, identifying a total of 26 noteworthy phase change materials (PCMs) within the temperature spectrum of 210–270°C. However, these materials were subjected to selection criteria in light of their associated health risks, the thermal and cycling stability of the materials. Two materials, Myo-inositol and solar salt, culminated in the final phase of the selection process. Moreover, Myo-inositol underwent thermal cycling within a closed system; however, it exhibited instability after 50 cycles, thereby designating solar salt (40 wt % KNO3/60 wt % NaNO3) as the uniquely suitable material.

In the process of identifying the most appropriate phase change materials (PCMs) for thermal energy storage within the temperature interval of 120-150°C, Haillot *et al.* (2011) established and adhered to a set of material selection criteria predicated upon their toxicological properties and ecological ramifications; economic considerations (derived from laboratory procurement costs) as well as hygroscopic characteristics. An extensive array of PCMs, encompassing solid–solid transition materials, sugar alcohols, polymeric hydrocarbons, and aromatic hydrocarbons, among other substances, were evaluated against the established screening criteria, resulting in the identification of only 11 PCMs that met the requisite selection parameters. The phase change materials (PCMs) were systematically characterized utilizing differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) in conjunction with a quadrupole mass spectrometer (QMS). The analyses were conducted prior to and after multiple (5) thermal cycles, and findings indicate that only a few of the PCMs (organic ones) exhibit the potential for thermal energy storage applications. This necessitates further investigations into their long-term stability. Their investigation underscores the significance of the measurement conditions on the resultant data.

Miro et *al.* (2016) proposed an innovative methodology for assessing the appropriateness of a phase change material (PCM) for thermal energy storage. The methodology involves a comprehensive enumeration of properties to be evaluated during the selection process of a PCM. It integrates considerations of health hazards, as well as both cycling and thermal stability, as essential criteria that must be addressed when identifying a suitable PCM for a specific application. The paper underscores the significance of health hazards, which is crucial both for the implementation of health and safety protocols (aimed at safeguarding employees who interact with phase change materials (PCMs)) and for the design and upkeep of systems. Similarly, cyclic behavior and thermal stability investigation yield valuable insights into their durability, which is a critical consideration when determining a suitable material for a specific application. To substantiate the experimental methodology described in this work, five distinct PCMs from various categories within the 150-200°C temperature spectrum were identified. They include salicylic acid, d-mannitol, hydroquinone, benzanilide as well as potassium thiocyanate. Comprehensive assessments of melting points, enthalpies, potential health risks, alongside cycling and thermal stability for both open and closed systems are conducted to a mass the essential data for a precise PCM selection. Benzanilide and d-mannitol were found out to be suitable in closed systems.

Gasia *et al*. (2017) conducted an investigation into the suitableness of 16 PCMs for subsequent utilization within a pilot plant experimental framework. This framework is designed to investigate the performance of a thermal energy storage (TES) system integrated with thermal processes operating within a temperature spectrum of 120 to 200°C, specifically under partial load conditions. The selection criteria employed were based on the parameters established by Miró *et al*. (2016). Nevertheless, the assessment of health hazards was broadened through the application of an additional standard classification, and the number of thermal cycling & analysis was increased to 100 cycles under an atmospheric environment that emulates the boundary conditions of the pilot plant's experimental framework while utilizing a greater sample mass. The study ultimately determined that adipic acid and high-density polyethylene (HDPE) emerge as the most viable candidate PCMs. These materials do not exhibit losses exceeding 12% of their thermal storage capacity after 100 cycles, do not manifest significant alterations in their chemical structure, and demonstrate low toxicity values, with degradation commencing at temperatures above 200°C.

Most of the literature was limited to low and medium temperature range (less than 200^{o}C) and to the best of our knowledge there is a lack of work presented for a higher temperature spectrum. The focus of this study is to identify the most appropriate phase change materials (PCMs) for the effective storage of thermal energy within the temperature spectrum of 200-400°C. The focus on this temperature range is that for solar cookers to replace conventional cooking methods they must be able to perform all range of cooking including high temperature such as baking and frying. Inorganic materials known for their high melting temperature will be considered in the course of this study. In doing so a similar PCMs screening approach established in literature by Haillot et al., 2011 and Maldanado et al., 2018 was adopted. Furthermore, this study will investigate the effect of storage containment (aluminium and stainless steel) on the degradation characteristics of the PCM. Most studies focus on the corrosion of the storage containment

MATERIALS AND METHODS

To select the appropriate materials, the current work reviewed literature to find potential PCMs in the temperature range between 200-400^{o}C. Twenty-nine inorganic PCMs materials (Salts and Eutectics) PCMs were found out to have melting temperatures in the temperature range being considered. For the study 98% purity KNO_3 and >99% purity $NaNO_3$, NaCl >99% purity and>99% purity KCl (Table 1) were acquired from Sigma Aldrich. Eutectic mixtures were prepared based on their weight percent as determined by FactSage software adopted from a related previous work (Gomez, 2011) using powder mill machine. Both the single and eutectic mixture materials were pre-dried for 24 hr at 110^{o}C to remove moisture content before analysis.

Table 1: Inorganic Single Component PCMs With Reported Melting Temperature 200-400^{o}c

Single Component Materials			
S/N	**Material**	**T_m (^{o}C)**	**Source**
1	$LiNO_2$	222	Kenisarin, (2009)
2	$LiNO_3$	253	Kenisarin, (2009)
3	$NaNO_2$	270	Kenisarin, (2009)
4	$ZnCl_2$	280	Xu *et al*., (2015)
5	$RbNO_3$	305	Muhammad, (2018)
6	$NaNO_3$	306	Gomez, (2011)
7	NaOH	317	Xu *et al.* (2015)
8	KNO_3	335	Gomez, (2011)

Single Component Materials			
S/N	**Material**	**T_m (°C)**	**Source**
9	CsOH	342	Muhammad, (2018)
10	$PbBr_2$	371	Muhammad, (2018)
11	KOH	380	Mohamed *et al*. (2017)

Table 2: Inorganic Multi-Component PCMs With Reported Melting Temperature 200-400°c

Binary Eutectic Mixture			
S/N	**Material**	**T_m (°C)**	**Source**
1	LiOH(30)–70NaOH	210–216	Kenisarin, (2009)
2	80 wt % NaOH/20 wt % LiOH	215	Maldonado *et al*. (2018)
3	40 wt % KNO_3/60 wt % $NaNO_3$	222	Majó *et al*. (2024)
4	KCl-$ZnCl_2$	230	Mohamed *et al*. (2017)
5	$NaNO_2$(80)–20NaOH	232	Kenisarin, (2009)
6	61 wt % NaOH/39 wt % $NaNO_2$	232-265	Kenisarin, (2009)
7	$NaNO_2$(27)–73NaOH	237	Kenisarin, (2009)
8	$NaNO_3$(72)–28NaOH	247	Kenisarin, (2009)
9	$NaNO_3$(18.5)–81.5NaOH	257	Kenisarin, (2009)
10	LiCl(37)–63LiOH	262	Kenisarin, (2009)
11	$NaNO_3$(41)–59NaOH	266	Kenisarin, (2009)
12	KNO_3 (90)/KCl	308.4	Umar *et al*. (2021)
13	46.3Mg–53.7Zn	340	Kenisarin, (2009)
14	NaCl-KCl	360	Nazir *et al*. (2018)
15	96Zn–4Al	381	Mohamed *et al*. (2017)
Ternary Eutectic Mixture			
16	NaCl(7.8)–6.4Na_2CO_3–85.5NaOH	282	Nazir *et al*. (2018)
17	$CaCl_2$(10.37) KCl (53.11) LiCl (36.52)	338.36	Nazir *et al*. (2018)
18	Li_2CO_3(31)–35K_2CO_3–Na_2CO_3	397	Gomez, (2011)

Preparation of Eutectic Mixture

The eutectic mixture of potassium and sodium nitrate of 50g with chemical composition of 40% KNO_3 and 60% $NaNO_3$ predicted using factSage software (Gomez, 2011) was prepared by measuring 20g KNO_3 and 30g $NaNO_3$ using an analytical weighing machine. After weighing, the samples were placed inside an oven for 24hrs at 110°C to remove moisture that may be absorbed during weighing process. The samples were removed from the oven and the mass of the sample was confirmed using analytical weighing machine to note any loss during drying. Afterward, the eutectic mixture was prepared by mixing the individual samples based on 40: 60 KNO_3 and $NaNO_3$ ratio. DSC machine was used to determine the melting temperature and enthalpy of the mixture. However, DSC analysis requires a few milligrams of the sample leading to the need of a homogenous mixture of the samples. To achieve that Retsch MM 500 Mixer Mill with a frequency of 30 Hz and 15-30 mins processing time was used for a through mixing of the individual samples. The same procedure was applied for the eutectic mixture of NaCl/KCl. FTIR analysis was conducted to ascertain the eutectic mixtures formed through their different functional groups.

Health, Fire Hazard and Chemical Instability

Detailed information on the risk possess by PCMs is crucial to establish procedures and precautions while handling such material. Therefore, in the course of this work SDS data of the PCMs through National Fire Protection Association NFPA ‘704’ (Fire Diamond) standard was adopted. NFPA is a universally accepted standard for safety that promote safety standards, education, and advocacy on fire and electrical related hazards. Any suitable PCMs must strictly adhere to the standard set by NFPA ‘704’ (Fire Diamond). This standard visually provides the riskiness of common chemical products by means of a colored diamond. Figure 1 presents the NFPA ‘704’ classification of hazardous materials. The diamond is divided in four indicators: flammability, health hazard, chemical reactivity, and special hazards. The figure provides the ranking from 0 to 4 for each indicator where 0 is the least while 4 is the highest under each category. For example, under health hazard, 0 = non-hazardous, 1 = slightly hazardous, 2 = hazardous, 3 = extreme danger and 4 = material that could cause death or major residual injury by very short exposure.

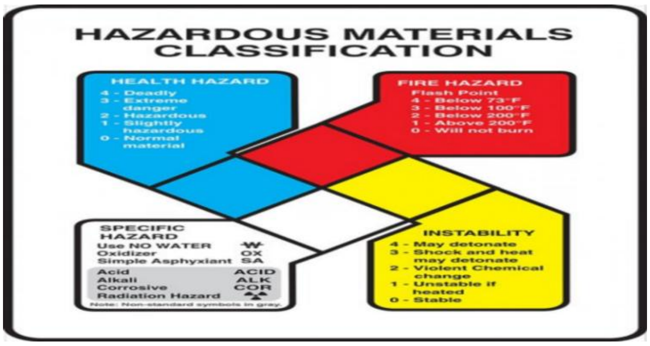


Figure 1: National Fire Protection Association (NFPA) '704' Diamond Standard (Maldonado *et al.*, 2018)

In the screening of the PCMs the following were considered under each hazard indicator:

i. Health Hazard: Materials that fall under class 0 to 2 were considered since the risk can be tackled by using PPEs while those materials in 3 and 4 were rule out.
ii. Fire Hazard: Materials under class 0 and 1 were considered, this is so because the materials in such class will not burn under the operating temperature range hence materials classified from 2 to 4 were selected out.
iii. Instability: Materials with class 0 were considered, since the application here involves heating the material.

Latent heat of Fusion

Appropriate PCMs are required to exhibit a relatively high latent heat of fusion to fully exploit their potential benefits of high energy density. An ideal PCM ought to possess a heat of fusion exceeding 100 J/g (Gomez, 2011). Consequently, substances with a latent heat of fusion greater than 100 J/g were taken into consideration. All substances with a latent heat of fusion below this threshold were excluded from the present investigation. However, materials with latent heat of fusion below this value can be considered for other applications.

Economic (laboratory supply-based price) and hygroscopic nature

Economic consideration is important in selecting materials for thermal energy storage (TES). This will ensure that TES systems are not only technically feasible but also financially viable, promoting broader adoption and long-term sustainability of PCMs. Materials with lower costs can make TES systems more affordable and increase their adoption particularly in solar cookers application. The cost of the PCMs in relation to energy stored were determined through the supply price and the enthalpies of the PCMs. Hygroscopic nature of a material is crucial in the selection of materials for thermal energy storage (TES). Hygroscopic substances possess the capacity to uptake moisture from the surrounding atmosphere, a phenomenon that may result in alterations to their thermal characteristics, consequently leading to inconsistent performance and reduced efficiency in energy storage applications. Thus, selection of weekly hygroscopic PCMs ensures stable, efficient, and safe thermal energy storage systems. Therefore, any material that is high in cost and highly hygroscopic in nature was discarded in the present work.

Chemical Structure and Thermophysical Properties Characterization

The chemical characterization was conducted employing Fourier transform infrared (FT-IR) spectroscopy in conjunction with attenuated total reflectance (ATR), which examines the chemical structure of the PCMs. ATR offers the benefit of acquiring and analyzing (both solid and liquid) sample spectra directly without requiring sample preparation (Fern *et al*., 2018) and samples both in solid and liquid can be easily analyzed. It works with a wavelength range between 4000 and 650 cm^{-1} with a standard spectral resolution of 4 cm^{-1} accounting for 64 infrared scans for each analysis. This analysis was carried out with a PIKE MIRacle™ ATR sampling accessory with a Diamond/ZnSe ATR base, FT-IR 6300 (Hachioji, Tokyo, Japan). The values obtained are the means. The outcome is determined by the characteristic wave numbers at which the molecules vibrate in infrared frequencies.

The melting point and latent heat of fusion of the chosen PCMs were determined using a differential scanning calorimeter (DSC) machine with standard reference material (alumina, Al_2O_3) and constant heating rate (10°C/min) under air atmosphere. The sample and reference holder, heater, heat resistor, and heat sink make up DSC (Figure 2). Through a heat sink and a heat resistor, the heater's heat is transferred to the sample and the reference. The heat differential between the heat sink and holders determines the amount of heat transfer. The temperature differential between the two holders determines how much heat is transferred to the sample and the reference. The DSC curve is a plot of temperature and heat flow. The melting temperature is estimated by the tangent at the point of greatest slope on the peak's face portion, and the latent heat of fusion is calculated using the area under the peak (Sa'eed, 2016).

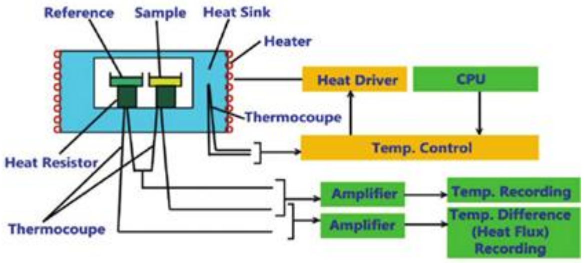


Figure 2: A Typical DSC Machine (Saeed, 2016)

Transient Hot-Bridge technique was adopted using a thermal conductivity measuring instrument (THB1, Linseis Inc., USA). A kapton insulated sensor (Sensor/B/MFR) equipped with enforced metal frame with a length of 105 mm and width of 42 mm was used to measure the thermal conductivity. A constant current was passed through the wire, generating heat that radiates outward and the rate of the temperature increase in the wire is inversely proportional to the thermal conductivity of the surrounding material. Calorimeter was used to calculate the specific heat capacity experimentally. As was previously shown in (Xiao *et al*., 2014), this study also computed thermal diffusivity mathematically using the density, thermal conductivity, and specific heat capacity relation.

Thermal Cycling and Chemical Stability Analysis

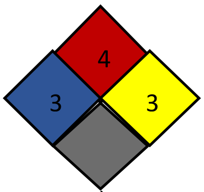
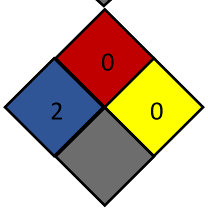
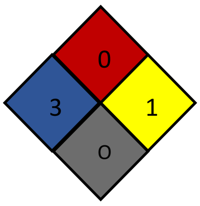
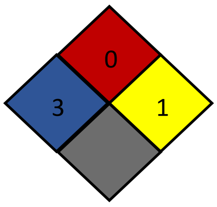
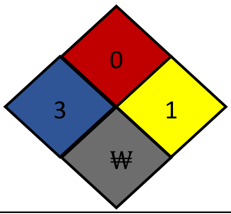
After a specific number of melting and freezing cycles, the cycling stability test is used to examine changes in PCM's thermophysical and chemical characteristics. An oven with model number NGH9240 from NANBEI Instrument Limited, China with a constant heating/cooling rate (10^{o}C /min) was used in the current study's cyclic stability tests. Samples of 10 g of each material were enclosed in an open ceramic crucible under air atmosphere. To achieve complete melting of the sample the temperature was set 10^{o}C above the respective individual melting point of the samples. To analyze thermophysical properties using a DSC machine, a small sample (5 mg) of PCM was taken out every ten cycles during the 100 cycles of extensive cycling experiments. After 100 cycles, the chemical structure of the PCMs was examined using FTIR. The PCMs underwent 100 cycles of comprehensive cycling tests, with a tiny sample of PCM removed every 10 cycles to examine thermophysical properties using a DSC machine. Following 100 cycles, FTIR was used to analyze the PCMs' chemical structure.

RESULTS AND DISCUSSION

Pre-selection based on Health, Fire Hazard and Chemical Instability

Table 2a and b shows the health hazard characterization of the considered single and multi-component inorganic PCMs, following the NFPA '704' standard. PCMs were categorized as suitable and non-suitable based on their hazard rating. Overall results indicated that single component materials were less hazardous compared to the multi-component inorganic counterpart.

Table 3: Results Of Single Component PCMs Health Hazard Evaluation With Their Respective Melting Temperature Following NFPA '704' Standard (Figure 1)

S/N	Material	T_m (ºC)	Hazard rating, NFPA '704'	TES Suitability
1	$LiNO_2$	222		Not Suitable Highly hazardous material
2	$LiNO_3$	253		Suitable Required used of PPEs during handling and operation
3	$NaNO_2$	270		Not Suitable Highly hazardous material
4	CsOH	272		Not Suitable Highly hazardous material
5	$ZnCl_2$	275		Not Suitable Highly hazardous material

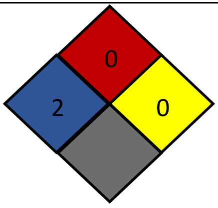
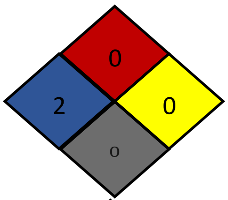
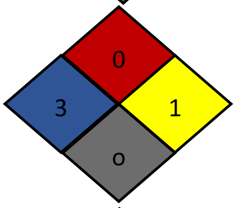
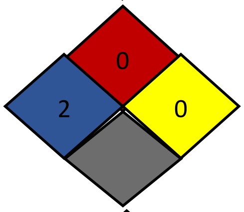
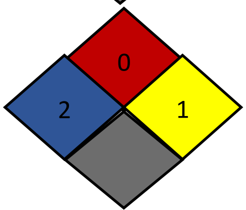
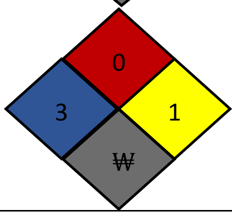
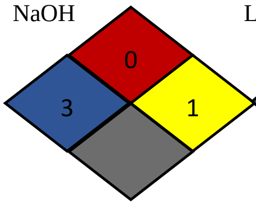
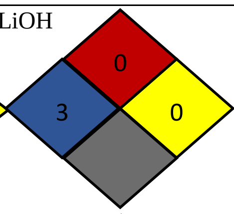
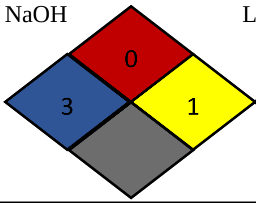
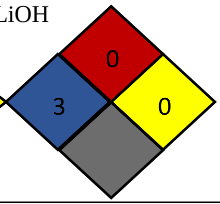
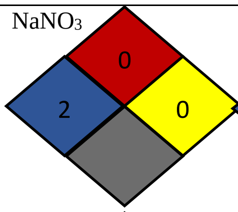
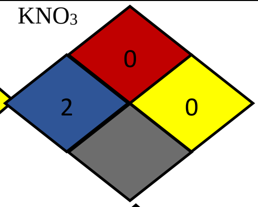
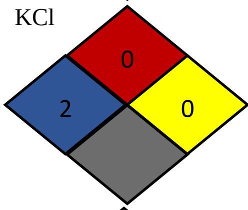
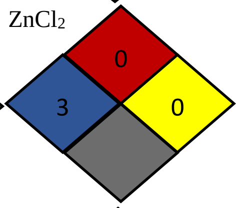
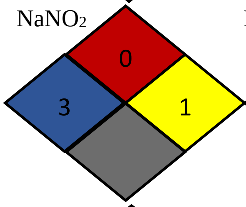
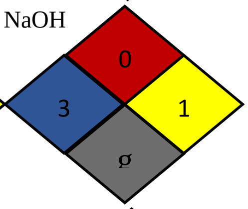
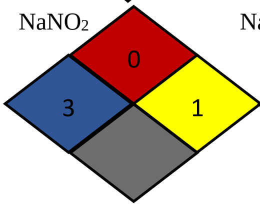
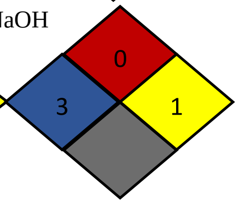
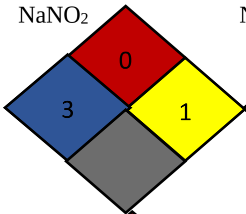
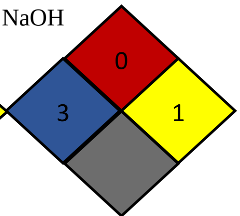
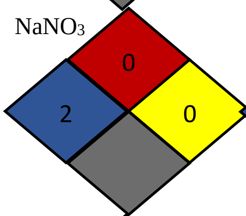
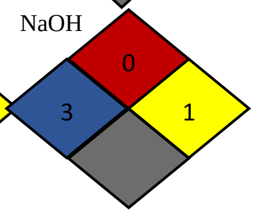
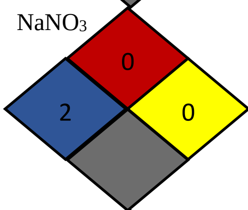
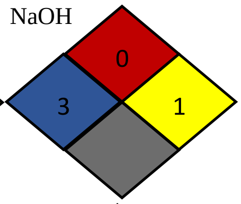
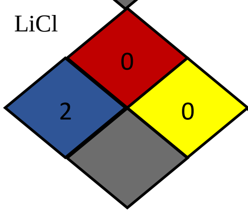
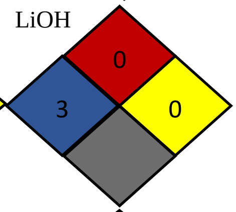
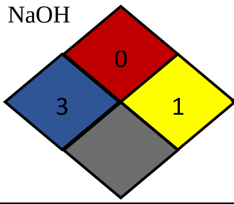
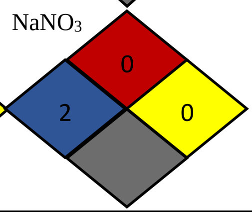
S/N	Material	T_m (°C)	Hazard rating, NFPA '704'	TES Suitability
6	$NaNO_3$	306		Suitable Required used of PPEs during handling and operation
7	$RbNO_3$	305		Suitable Required used of PPEs during handling and operation
8	NaOH	317		Not Suitable Highly hazardous material
9	KNO_3	335		Suitable Required used of PPEs during handling and operation
10	$PbBr_2$	371		Suitable Required used of PPEs during handling and operation
11	KOH	385		Not Suitable Highly hazardous material

Table 4: Results of Multi-component PCMs Health Hazard Evaluation with their Respective Melting Temperature Following NFPA '704' Standard (Figure 1)

			BINARY EUTECTIC PCMs		
S/N	Material	T_m (°C)	Hazard rating, NFPA '704'		TES Suitability
1	LiOH(30)–70NaOH	210–216	NaOH 	LiOH 	Not Suitable Highly hazardous material
2	80 wt % NaOH/20 wt % LiOH	215	NaOH 	LiOH 	Not Suitable Highly hazardous material

3	40 wt % KNO_3/60 wt % $NaNO_3$	222	$NaNO_3$ 	KNO_3 	Suitable Required used of PPEs during handling
4	KCl-$ZnCl_2$	230	KCl 	$ZnCl_2$ 	Not Suitable Highly hazardous material
5	$NaNO_2$(80)–20NaOH	232	$NaNO_2$ 	NaOH 	Not Suitable Highly hazardous material
6	61 wt % NaOH/39 wt % $NaNO_2$	232-265	$NaNO_2$ 	NaOH 	Not Suitable Highly hazardous material
7	$NaNO_2$(27)–73NaOH	237	$NaNO_2$ 	NaOH 	Not Suitable Highly hazardous material
8	$NaNO_3$(72)–28NaOH	247	$NaNO_3$ 	NaOH 	Not Suitable Highly hazardous material
9	$NaNO_3$(18.5)–81.5NaOH	257	$NaNO_3$ 	NaOH 	Not Suitable Highly hazardous material
10	LiCl(37)–63LiOH	262	LiCl 	LiOH 	Not Suitable Highly hazardous material
11	$NaNO_3$(41)–59NaOH	266	NaOH 	$NaNO_3$ 	Not Suitable Highly hazardous material

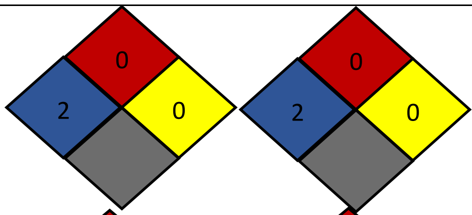
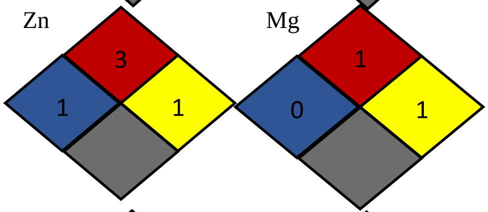
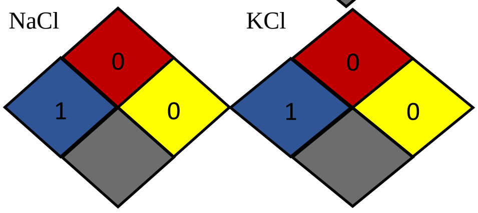
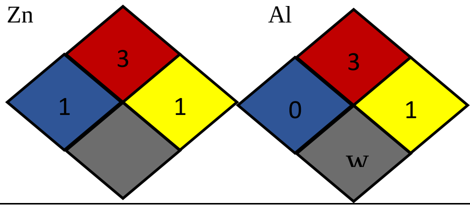
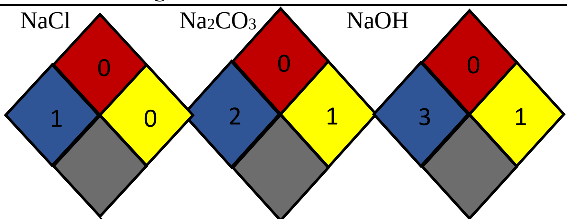
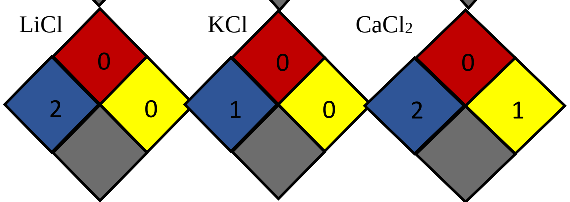
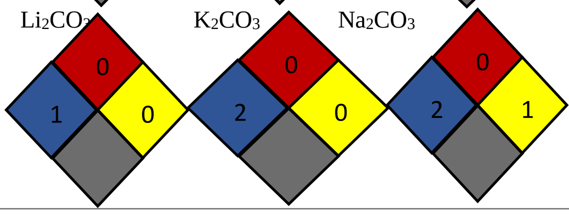
12	KNO_3 (90)/KCl	308.4		Suitable Required used of PPEs during handling
13	46.3Mg–53.7Zn	340	Zn Mg 	Not Suitable Chemically unstable when heated
14	NaCl-KCl	360	NaCl KCl 	Suitable Required used of PPEs during handling
15	96Zn–4Al	381	Zn Al 	Not Suitable Chemically unstable when heated

Table 5: Results of Component PCMs Health Hazard Evaluation with their Respective Melting Temperature Following NFPA '704' Standard (Figure 1)

TERNARY EUTECTIC PCMs				
S/N	**Material**	**T_m (°C)**	**Hazard rating, NFPA '704'**	**Suitability**
1	NaCl(7.8)–6.4Na_2CO_3–85.5NaOH	282	NaCl Na_2CO_3 NaOH 	Not Suitable Highly hazardous material
2	CaCl2(10.37) KCl (53.11) LiCl (36.52)	338.36	LiCl KCl $CaCl_2$ 	Suitable Required used of PPEs during handling
3	Li_2CO_3(31)–35K_2CO_3–Na_2CO_3	397	Li_2CO_3 K_2CO_3 Na_2CO_3 	Suitable Required used of PPEs during handling

A special safety study would be necessary if any of the PCMs with a higher hazard rating that were designated as unsuitable were chosen as ideal candidates. This could result in changes to storage system and therefore increases the cost of the system under consideration (Maldonado *et al.*, 2018). In the course of the present work single or eutectic mixtures of NaOH, KOH, LiOH, $LiNO_2$, $NaNO_2$, $ZnCl_2$, CsOH, Zn and Al marked as unsuitable will be discarded.

Screening Based on Latent Heat of Fusion

PCMs that satisfied the health hazard requirement were subject to further screening based on their respective latent heat of fusion. PCMs having enthalpy less than 100 kJ/kg are not good PCMs (Gomez, 2011) thus, were discarded. Figures 3 and 4 present the single and multi-component PCMs plotted against their respective latent heat of fusion.

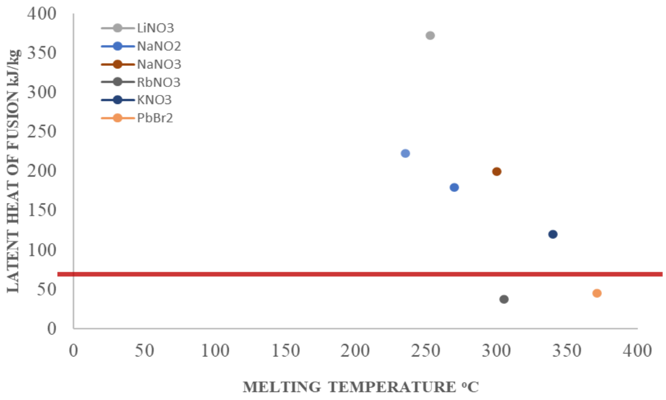


Figure 3: Single Component PCMs

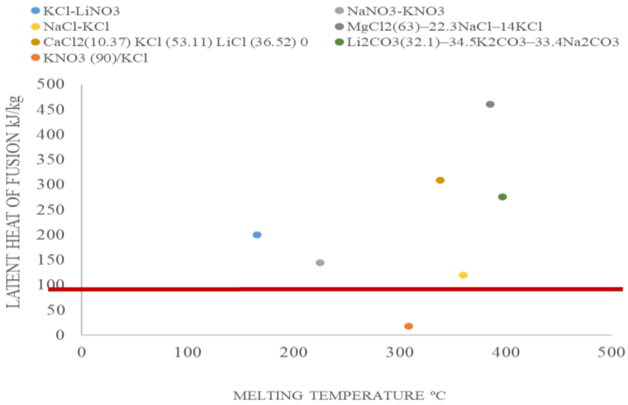


Figure 4: Multi-component PCMs

From the results obtained in Figures 3 and 4, two PCMs, $PbBr_2$ and $RbNO_3$, shows a latent heat of fusion below 100 J/g whereas eutectic PCMs shows a remarkably higher latent heat with only one candidate KNO3/KCl fall below 100 J/g. These materials presented low energy storage density as mentioned in the work of (Gomez, 2011) and therefore were discarded as this stage of screening.

Economic (Laboratory Supply Price) Screening

The PCMs that satisfied latent heat of fusion criteria were further screened based on the small-scale laboratory supply price. The cost by quantity can be seen in the figure 5.

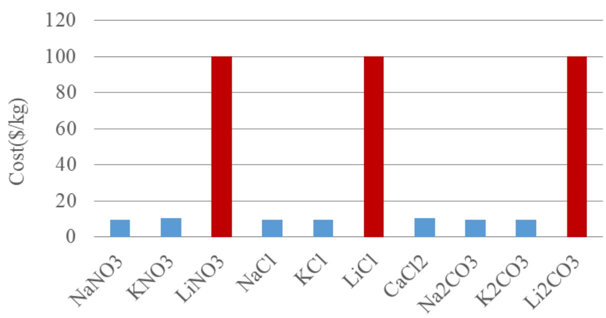


Figure 5: Cost of Materials ($/kg)

Based on cost per kilogram $LiCO_3$, LiCl and $LiNO_3$ show relatively very high cost (greater than 20 $/kg) compared to the other suitable PCMs. The high cost of these materials is due to the present of Lithium in the compounds with a price >150 €/kg and considering it as a PCMs for TES application will increase the cost of the TES system (Maldonado *et al.*, 2018) and therefore both single and multi-component materials having a lithium element were discarded.

Table 3: Pre-Selected Materials with Their Respective Melting Temperature and Enthalpies

S/N	PCMs	T_m (°C)	L (kJ/kg)	Reference
1	$NaNO_3$	306	172-187	(Maldonado *et al.*, 2018)
2	KNO_3	340	120	(Nazir *et al.*, 2018)
3	40 wt % KNO_3/60 wt % $NaNO_3$	210-220	108.67	(Foong *et al.*, 2011)

4	58 w% NaCl- 42 w% KCl	360	120	(Nazir *et al.*, 2018)

Chemical Structure and Thermo-physical Properties Characterization

The materials selected (Table 3) following the screening steps discussed in subsection 3.1 through 3.3, are characterized in terms of their chemical structures and thermo-physical properties.

Chemical Structures (FTIR)

PCMs chemical structures were identified using FTIR. This helps in identifying materials composition through it functional group. From the figure 6-8 it can be seen clearly that there are two major peaks from the results of the FTIR for Nitrate salts with peaks corresponding to the functional group of nitro compounds. Precisely, the presence of peak at approximately 1341-1365 cm^{-1} in all the three compounds ($NaNO_3$, KNO_3 and Solar salt) indicated the presence of N-O asymmetric stretching vibration and peak around 823-835 cm^{-1} indicated N-O out of plane bending (Orozco *et al.*, 2021).

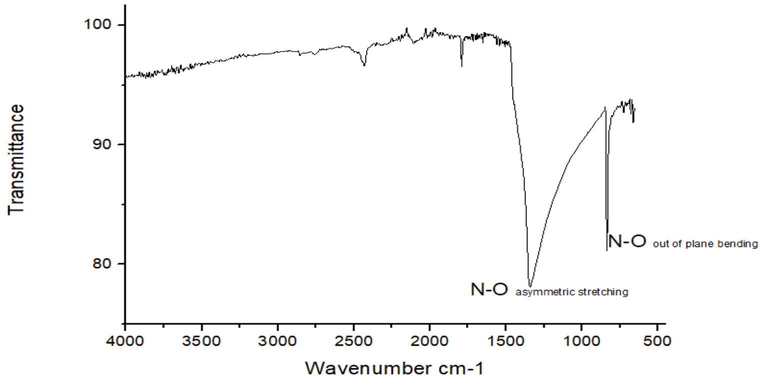


Figure 6: $NaNO_3$ FTIR Analysis Result (Standard Resolution of 4cm^{-1} and 64 Scans)

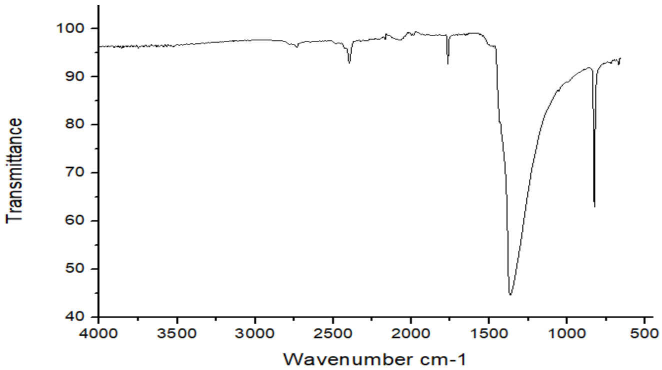


Figure 7: KNO_3 FTIR Analysis Result (Standard Resolution of 4cm^{-1} and 64 Scans)

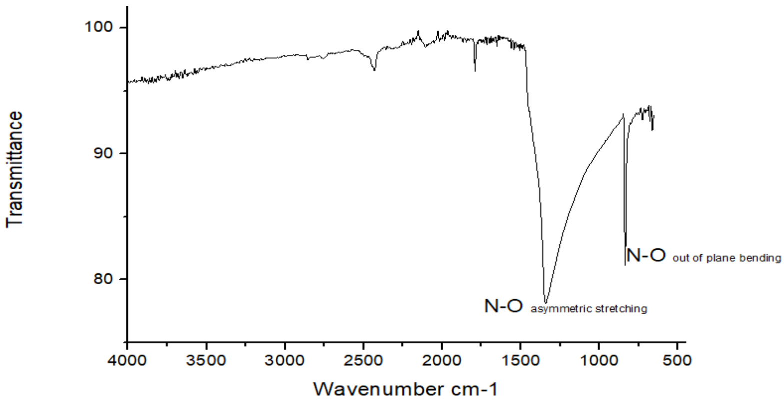


Figure 8: $NaNO_3$/KNO_3 (Solar salt) FTIR Analysis Result (Standard Resolution of 4cm^{-1} and 64 Scans)

Thermo physical Properties Characterization

DSC study was carried out for each PCMs that scaled the preselection stage to identify the (phase change) transition temperature and latent heat of fusion. Likewise, thermal parameters of the preselected PCMs including specific heat capacity (Cp), thermal conductivity (λ), and Density (ρ) were experimentally determined. The mathematical expression in

Equation 1 is used to compute the thermal diffusivity (α), as taken from (Saeed, 2016; Xiao *et al.*, 2014).

$$a = \frac{?}{?Cp} \qquad 1$$

Where;
Cp = Specific heat capacity
λ = Thermal conductivity
ρ = Density
α = Thermal diffusivity

DSC analysis was conducted for Sodium nitrate in order to determine its suitability for use as a thermal energy storage material. The DSC curve in Figure 9 showed that the latent heat of fusion and melting temperature of sodium nitrate were 171.4 J/g and 307 ^{o}C, respectively. The results obtained were compared with various similar work from literature. Michels and Roberts (2006) used DSC machine to measure the thermophysical characteristics of a few chosen PCMs and reported the melting temperature and the corresponding latent heat of fusion of $NaNO_3$ as were determined as 306 ^{o}C and I72 J/g respectively. From a different study, Umar et al. (2021) carried out a DSC test using dynamic mode and experimentally determined the melting temperature and latent heat of fusion of $NaNO_3$ as 305.9 ^{o}C and 175.9 J/g respectively. Similarly, other literatures (Gomez, 2011; Lomanaco et al., 2015; Kenisarin, 2009), agreed with the results obtained in the present study. Likewise, other literature sources (Gomez, 2011; Lomanaco *et al.*, 2015; Kenisarin, 2009) corroborated the findings from the current study.

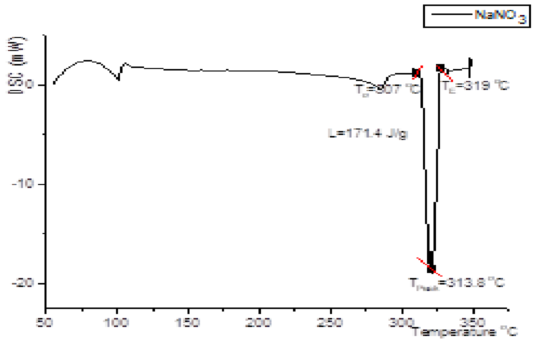


Figure 9: $NaNO_3$ DSC Thermogram

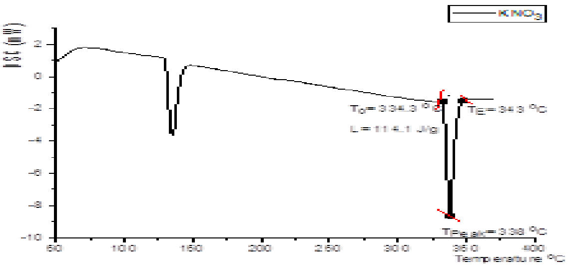


Figure 10: KNO_3 Thermogram

From Figure 10 the melting temperature and enthalpy of KNO_3 was experimentally determined using DSC as 334.3oC and 114.1 J/g respectively. The results are agreement with literatures (Chen and Wan, 2011; Jriri et al., 1995; Nazir et al., 2018; Orozco et al., 2021).

Figure 11 presented a result of $NaNO_3/KNO_3$ where enthalpy and melting temperature of mixture were obtained as 110 J/g and 219^{o}C respectively. Kanasarin (2009) reported 117 J/g and 222^{o}C as enthalpy and melting temperature of $NaNO_3/KNO_3$ respectively. Similarly, Maldonado et al. (2018) work on PCM selection for thermal energy storage within 210^{o}C to 270^{o}C range of temperature, and reported enthalpy of 94.5 J/g as well as a melting temperature of 220.8^{o}C using DSC for $NaNO_3/KNO_3$. The slight difference in the enthalpy values observed maybe due materials purity or homogeneity of the prepared eutectic mixture.

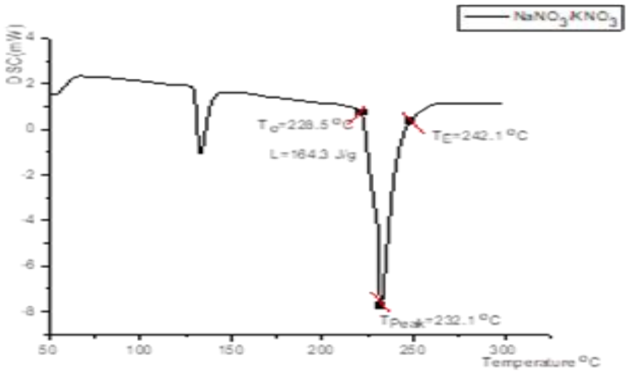


Figure 11: $NaNO_3$/KNO_3 Thermogram

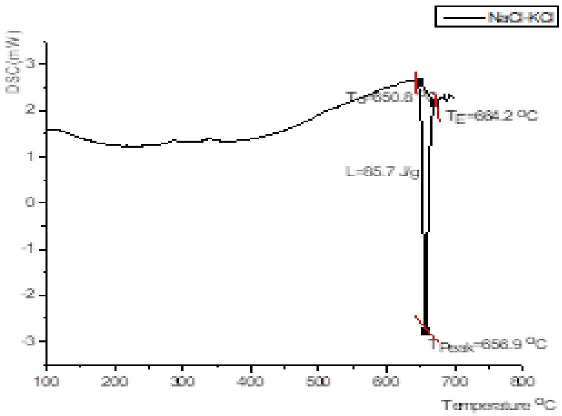


Figure 12: NaCl/KCl Thermogram

For NaCl/KCl mixture, the melting temperature was found to be 650.8 °C while enthalpy was determined as 85.7 J/g (Figure 12). However, the results obtained differ with the literature values. Nazir *et al*. (2018) determined enthalpy (of the NaCl/KCl mixture) as 120 J/g with a melting temperature of 360 °C. Similarly, Xu and Chan (2015) reported enthalpy and melting temperature of eutectic material (58 w%) NaCl/ (42 w%) KCl as 119 J/g and 360 °C respectively. However, Tianying et al. (2021) and Lonergan et al. (2023) reported a melting temperature of between 600 and 800°C. This makes this PCM not suitable in the temperature range for Solar Cookers. There is the need for further study on this mixture and also chlorides have high corrosion and thus for them to be suitable the corrosion behavior has to be improved. Thus further analysis of this material was not conducted.

With regards to other thermo physical properties, Michels and Roberts (2006) in their work reported the density of $NaNO_3$ and KNO_3 as 2261 and 2109 kg/m^3 respectively, the same work reported a thermal conductivity of both the PCMs as 0.5 W/m.K and similarly the Cp of $NaNO_3$ and KNO_3 were reported as 1.10 and 0.953 kJ/kg.K These values reported corresponded with the values obtained in current work (Table 4).. Xiao *et al* (2014) syntheses a mixture of the so called solar salt $NaNO_3$ and KNO_3 (6:4) and reported determined the Cp, thermal conductivity and thermal diffusivity as 2.351 kJ/kg.K, 2.272 W/m.K and 0.477 mm^2/s and the density was calculated using the mathematical relation. These values agreed with the ones determined in the present work (Table 4).

Table 4: Density, Specific Heat Capacity, Thermal Conductivity and Diffusivity of the PCMs

S/No	Sample	Density, ρ (kg/m$^{3)}$	Specific heat capacity, Cp, (J/g.K)	Thermal conductivity λ, (W/m.K)	Thermal diffusivity, α, (mm^2/s)	Reference(s)
1	$NaNO_3$	2,242	1.374	0.6099	0.1980	Present work
		2,261	1.10	0.50	-------	(Michels and Pitz-paal, 2006)
2	KNO_3	2,205	0.938	0.4176	0.2019	Present work
		2,109	0.953	0.50	-------	(Michels and Pitz-paal, 2006)
3	$NaNO_3$-	1,886	2.572	0.580	0.1196	Present work
	KNO_3	1,889	2.351	2.272	-------	2016)

Thermal Cycling, Chemical Stability and Containment Compatibility

Thermal cycling stability test was conducted to check alterations in the thermophysical characteristics of the PCM after experiencing several solidification-melting cycles within the operational temperature range of the process where the TES material will be utilized. The current study seeks to find a suitable PCM for solar cooking application and therefore, the substance known as 'solar salt' which has met the screening requirements and exhibit good thermophysical properties was further analyzed to study it long term application. The solar salt with stainless steel and aluminum inserted inside to check their compatibility as the storage container was exposed to 100 complete solidification-melting cycles.

The results related to the melting temperature and enthalpy of the solar salt obtained through DSC at a heating rate of 10 °C/min are presented in Table 5. After 20, 50 and 100 cycles samples were taken for the DSC test, and the test was conducted with three samples in-order to get a result representing bulk behavior since the DSC test requires very small sample size.

Table 5: Melting Temperature and Enthalpy of PCMs from 0-100 Thermal Cycles

Solar salt with stainless steel			**Solar salt with aluminum**		
Number of cycles	**Melting temperature (ºC)**	**Latent heat of fusion (J/g)**	**Number of cycles**	**Melting temperature (ºC)**	**Latent heat of fusion (J/g)**
0	219	110	0	219	110
20	217.8	107.2	20	222.3	90.8
50	213.3	105.1	60	215.4	85
100	216.8	97.1	100	225	65

The melting temperature and enthalpy of the PCMs were measured prior to and following thermal cycles. As indicated in the results shown in Table 5, the alterations in melting temperature and enthalpy rise with the increasing number of cycles. From the table it will be observed that the melting temperature is relatively stable and the change in the latent heat of fusion after 100 cycles is about 11% which shows little degradation over the 100 cycles. The discrepancy in the latent heat of fusion over different cycles can be attributed to the formation of metastable phases which were recently explored by Sergeeve *et al.*, (2025). It was identified that metastable solid solutions are formed upon cooling of the PCM and thus suggested the use of advanced techniques for characterization. Maldonado et al. (2018) studied some selected PCMs and 50 thermal cycles were performed and reported a mixture $NaNO_3/KNO_3$ exhibited good thermal properties with an enthalpy of 94.7 J/g and melting temperature 222.2ºC. The results of PCMs consisting of stainless-steel piece obtained in the current work after 100 thermal cycles agree with reported values maintaining a stable latent heat 97.1 J/g with an acceptable 5% change in melting temperature. However, high loss presenting over 60% in latent heat compared was observed of the PCMs with aluminum piece inserted into it. Looking at the result for Solar Salt with Aluminium, it will be observed that from the 20th cycle there is a large change in the latent heat of fusion signifying massive degradation which may be due to the interaction between the Solar salt and aluminium which will require further analysis later in this work. The result obtained presented a good advantage of choosing stainless steel over aluminum as the storage material while using solar salt as PCMs for thermal energy storage.

To further understand the behaviour, FTIR tests were conducted on the Solar Salt (Eutectic mixture of $NaNO_3/KNO_3$) at 0th cycle and 100th cycle and comparison was made. Figure 13 presents the comparison of the FTIR results at 0th and 100th cycle for both with stainless steel and aluminium. The result showed that there was no chemical degradation as there are no changes observed in the absorption bands of before and after cycling test of the material after undergoing 100 cycles of melting/solidification as can be observed in Figure 13. Both N-O asymmetric and N-O out of plane bending remained unchanged and no new functional group observed making the mixture chemically stable after 100 thermal cycles. This has thus further extended the study of Maldonado *et al*. (2018) who reported that a mixture $NaNO_3/KNO_3$ was chemically stable after performing 50 thermal cycles.

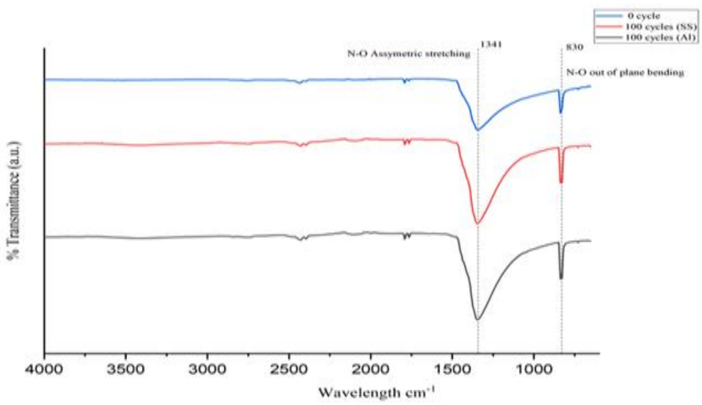


Figure 13: FTIR Analysis for $NaNO_3/KNO_3$ Eutectic Mixture at 0 and 100th Thermal Cycle with Stainless Steel and Aluminum

The degradation of the Solar Salt in Aluminium can be attributed to the mechanism of basic fluxing, in which the molten solar salt at temperatures around 300 °C dissolves the protective alumina scale layer which leads to aggressive and rapid progression of corrosion. This degradation not only results in significant material weight loss and the formation of aluminates, but it also releases metallic impurities that accelerate the thermal decomposition of the salt into nitrites and nitrogen oxides. This has been reported by Ruotong *et al.* (2025) and Paon *et al.* (2025) for high temperature applications. This thus means that aluminium containment is not suitable for solar cooker applications requiring high temperatures.

On the other hand, the Solar Salt is stable for several cycles and is compatible with stainless steel since it does not show a high discrepancy over several cycles. The discrepancies observed may be due to the errors in the experiment and the fact that DSC uses small samples and it all falls in what has been reported in the literature. This means that Solar Salt is a suitable PCM for high temperature solar cooking applications and this study has extended the thermal cycling study up to 100 thermal cycles which further provides confidence on this PCM.

CONCLUSIONS

The work extensively reviewed inorganic PCMs including eutectic mixtures for TES application, of which 29 potential PCMs were found out in the operating temperature range 200-400°C which is suitable for high temperature solar cooking. These PCMs were subjected to preselection screening criteria of health hazard, energy storage capability, and economics. Four (4) PCMs ($NaNO_3$, KNO_3, $KNO_3/NaNO_3$ and NaCl/KCl) satisfied the initial pre-selection criteria. Most of the PCMs failed the health hazard criteria.

Chemical structure and thermophysical properties characterization of the four (4) PCMs were carried out. Though some literature reported that NaCl/KCl eutectic mixture has a melting point of 360°C, this study repeatedly obtained a melting temperature of around 650°C which is corroborated with findings in some recent articles. Thus three (3) materials $NaNO_3$ and KNO_3 and solar salt (eutectic mixture of $KNO_3/NaNO_3$) satisfied this screening stage and showed remarkable properties making them suitable materials for TES application.

Thermal cycling and chemical stability analysis was then further conducted on the solar salt, also considering compatibility with Aluminum and Stainless Steel. Results showed that at these high operating temperatures aluminum interacts with PCM and cause massive degradation in the latent heat of fusion after 100 cycles. Stainless steel shows compatibility with solar salt and is recommended as containment for PCM. The thermal cycling indicated the suitability of solar salt for a long-term TES application in solar cookers with stainless steel as a storage container.

It is recommended that future work should critically look at the eutectic mixture of NaCl/KCl to confirm its melting temperature and determine whether different eutectic mixtures exist with different thermophysical properties. Further thermal cycling analysis beyond 100 thermal cycles can be conducted to ascertain the long-term stability of these screened PCMs. There is the need to develop novel materials that can serve as PCMs to be able to have a sustainable solar cooking system that can replace conventional cookers.

List of Abbreviations

ATR: attenuated total reflectance
Cp: Specific Heat Capacity
DSC: Differential Scanning Calorimeter
LHS: Latent Heat Storage
FTIR: Fourier Transform InfraRed
HDPE: High Density PolyEthylene
NFPA: National Fire Protection Association ‘’
PCM: Phase Change Material
QMS: Quadrupole mass spectrometer ()
TES: Thermal Energy Storage

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