DEVELOPMENT OF ALKALI CHLORIDES AS HEAT TRANSFER FLUIDS WITH ADDITION OF NANOPARTICLES

by

SRIRAM SAMBASIVAM

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This work is a dedication to my parents (M Sambasivam and S Bavani), who have always been there for me in all my adventures, my elder brother and sister in law (Mahadevan and Madhumitha), who helped me in my transition from India to USA and finally to all my friends here, who made my life enjoyable

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Abstract

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Sriram Sambasivam, MS

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Supervising Professor: Donghyun Shin

Recent studies have shown that using a single fluid for Heat Transfer and Thermal Energy Storage, improves the working and efficiency of a CSP. It improves the conversion efficiency (thermal energy to electrical) and also reduce the thermal losses due to heat exchangers by eliminating them. Other methods of storage are thermo chemical TES or phase change TES, but they require heat exchanger between HTF and TES medium, due to which the thermal losses between them are significant. CSP works on a conventional thermodynamic cycle (Rankine Cycle, Brayton Cycle) indicating that higher operating temperature are required to achieve higher efficiency. Higher temperature would also help us to use these materials in other applications like Oil refineries, molten salt reactors. However, lack of available fluids at higher temperatures are a hindrance towards raising the temperature. Conventional materials used as storage material are not thermally stable at higher temperature. The critical temperature of water 374.15^{o}C at 221.2 bar and organic storage fluid such as oil, ethylene glycol well below 400^{o}C. Molten salts can be an effective alternate material due to its stability at higher

temperatures and cheap availability. The upcoming solar thermal plants use eutectic fluid system (eg solar salt, $NaNO_3$-KNO_3) as TES material at higher temperature. This can raise the operating temperature from 300^oC to 500^oC as they start to decompose around that temperature. In order to further increase the operating temperature to around 700^oC, we need to look out for other materials. Alkali chlorides provide a suitable alternative as they are stable at this temperature. Along with low vapor pressure and good heat transfer characteristics, we can use them to store energy at higher temperature. The disadvantage of using them at higher temperature are its corrosion effect and its poor thermal properties. At higher temperatures ceramic or high-temperature alloys should be used as the structure materials due to the creep of stainless steel and therefore corrosion may not be an issue. Thermal properties can be enhanced by doping the base salt with nano particles. In this study, we have developed a binary eutectic chloride mixture (Lithium Chloride and Potassium Chloride) with addition of silicon oxide nanoparticles (2% weight addition) which show around 15% enhancement in its thermal heat capacity. This study would help in commercialization of these HTF and also effectively reduce the cost of production of electricity.

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Chapter 1

Introduction

1.1 Introduction to Solar Energy

Solar thermal Energy is a major sources for renewable source of energy. Due to limitations of fossil fuels and its effect on the environment, solar energy is of great significance. In general, solar energy deals with conversion of the solar energy from sun to other useful forms like thermal and electrical energy. It is also one of the cleanest and most abundant renewable energy source available. There are several ways in which we can harness this energy and it can be classified in two parts – Active Solar technique and Passive Solar technique. In case of Active solar technique, we convert the solar energy into other useful energy like electrical using other auxiliary systems. The prominent methods of Active Solar Techniques are Photovoltaics, and Concentrated Solar Power [1]. Similarly in case of Passive Solar techniques, we can directly use the solar energy to store and distribute to maintain the comfort and not involve any moving parts. Let us take a closer look at the Active solar techniques.

1.2 Photovoltaic Technology

Photovoltaic cells are basically semiconductors which convert the incident solar energy into electricity directly [1]. They are made of P and N type of semiconductor. When the cell absorbs the incoming solar radiation, due to the semiconductor generates electricity, which is transferred using an electrical circuit. The advantage of this technology is easier conversion of solar energy, modular assembly and easy to scale up the production. But the disadvantage of this technology is the cost of production of electricity and limitation in operational time [2]. We can only use this technology during the presence of sunlight and when it is not present, the production of electricity is not possible. This limits the application of Photovoltaics to a very large extent. In order, to overcome this we can use concentrated solar power plants.

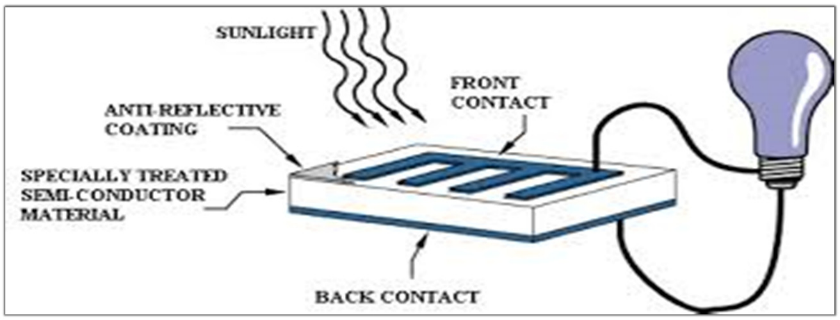

Figure 1.1 Photovoltaic [3]

1.3 Concentrated Solar Power Plant (CSP)

These plants use an array of mirror as optical elements to concentrate solar energy and convert them to thermal energy [4] . The general operating temperature of these plants are around 300°C – 600°C. Thus using CSP, the energy is converted into thermal energy, by heating the thermal storage material and then it is used to drive the generator which produces electricity. Commonly used CSP's are: Parabolic Trough, Power Tower and Dish Stirling.

1.3.1 Parabolic Trough

They are the most commonly and matured version of CSP present in the market. One most the recently erected in Arizona desert having a capacity of 280MW and another one in California for 354MW.

In general, all the parabolic troughs, contain sheets of material bent in the form of dishes and arranged in the form of a parabola. These act as the reflecting surface and are supported by pedestals from ground. They reflected the incident ray to a central receiver.

This central receiver is a black pipe coated with special materials that minimize the convective losses. HTF are running inside this tube, which absorbs the heat and stores it them. This is later on pumped to the generator to produce electricity. Most commonly used HTF's are mineral oil and water. They have high thermal properties and are less volatile upto 400°C. But in order to increase the operating temperature of the plant we need to use different heat transfer fluids.

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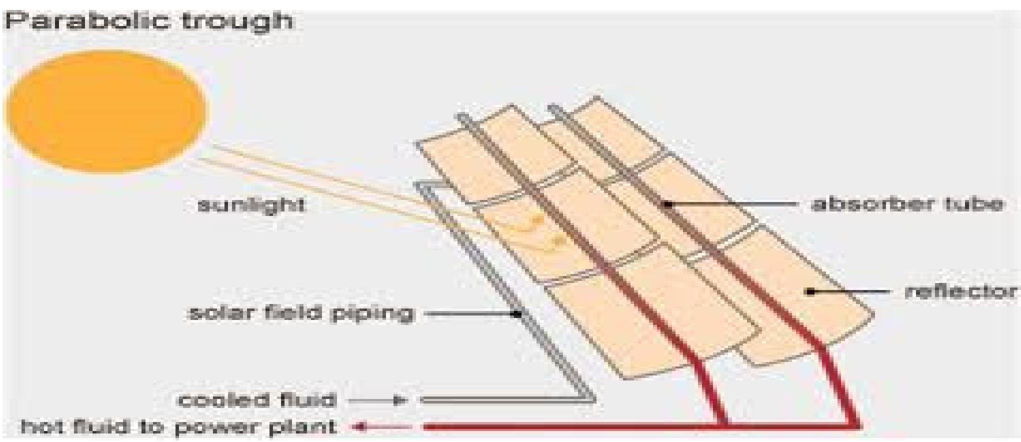


Figure 1.2 Parabolic Trough [5]

1.3.2 Power Tower

Power Tower system can be used for scaling up the solar energy plant capacity. The system consist of a large central tower with a receiver mounted on top of it. The ground level receivers consisting of mirrors direct the solar energy towards the receiver on top, from where the energy is transferred and stored to produce electricity [6].

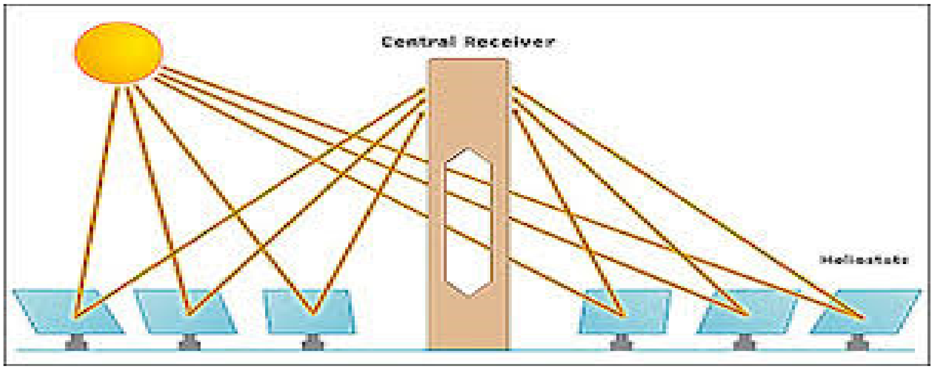


Figure 1.3 Central Tower CSP [7]

1.3.3 Dish Stirling

In case of a dish Stirling plant, a Stirling engine is mounted behind the receiver, thus directly converting the solar energy into mechanical energy [8].

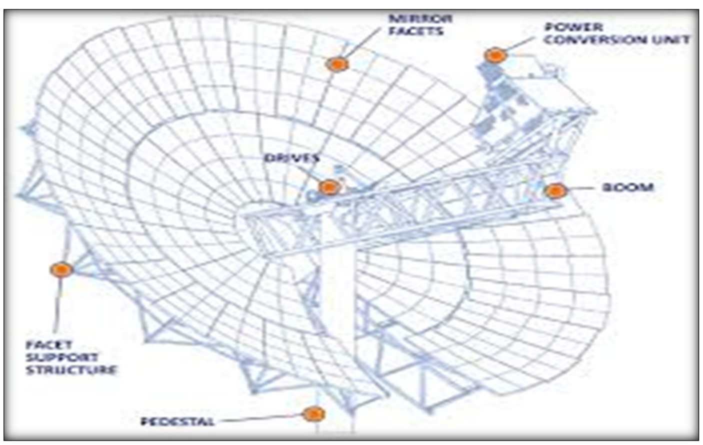


Figure 1.4 Dish Stirling CSP [9]

1.4 Working of CSP

Now that we have seen the different types of CSP, let's take a look at the working of one. A CSP plant works on a conventional Rankine Cycle for converting thermal energy into electrical. The major components of a CSP are

a. Solar Collector Field, which consist of an array of mirrors concentrating the incident solar power to the receiver.

b. Receiver

c. Heat Transfer Fluid Loop, consist of the fluid which absorbs the thermal energy from the receiver and transfers it to the Thermal Energy Storage (TES) system.

d. Heat Storage System, consist of TES tanks to store the energy and can be reused when the demand increases.

It can effectively said that we can improve the efficiency of the system, if we are able to reach higher operating temperature.

1.5 Common material for Heat Transfer and Thermal Energy Storage

As we have seen above that, HTF are responsible to transport the energy in the form of heat from the receiver to the TES system. Conventional HTF are mineral oil, paraffin wax etc., which have good heat transfer properties but have low decomposition temperature (<400oC) [10].

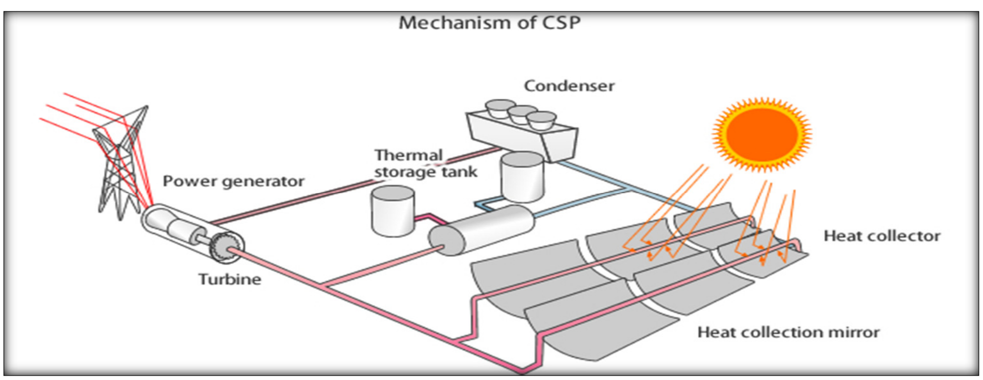


Figure 1.5 Working of CSP (Two Tank) [9]

The transferred heat from HTF are stored in TES system. It consist of storage tanks, which allows the storage of the produced thermal energy. Commonly thermal energy can be stored as: Sensible Heat, Latent Heat and Thermochemical Heat. Sensible heat storage can be done when we store energy due to the change in temperature of the material. Desirable properties for these material are less reactivity with the TES structure, stability with fluctuating temperatures and ideal vapor pressure [11]. Latent heat storage occurs when we store the thermal energy in the form of latent heat of fusion at the same temperature. The material provides higher energy density [12]. Both the storage methods have their own advantages and disadvantages.

TES also provides an additional support to the existing power plants as we can use the stored heat during peak hours. It can combined with any of the power generating unit to produce electricity [13]. It is due these advantages that we can use TES in CSP. Molten salts which are being considered as an alternated HTF can also be used for TES. This is very advantageous to us as this would eliminate the heat exchangers between the HTF and TES which in turn would reduce the thermal losses (exegetics losses). Moreover due to the low vapor pressure of molten salts, we require less pressure to pump them, as a result of which we can expect the reduction in the pumping cost. Another advantage in combining both are that we require less amount of salt as compared to the conventional plant .Overall we expect the efficiency of the plant to improve while the cost required for production of electricity would also come down [14].

1.6 Limitations of CSP

The significant limitation of CSP are in the overall cost of production of electricity as compared to the fossil fuel driven plants. As we have seen the major components of the plant are TES system, HTF and Heat storage material which contribute a lot towards the cost determination. By using molten salts, we can reduce these cost to a large extent [15]. We can increase the operating temperature of the system, thereby improve the efficiency. We can eliminate the heat exchangers between the TES material and HTF, thereby reducing the thermal losses and also reduce the cost. Similarly, we can reduce the pumping costs required for the heat transfer loop due to low vapor pressure of the salts [16]. Since the cost of molten salts are also very low as compared to the conventional fluids, we can bring down the cost considerably.

1.7 Improvement of Efficiency of CSP

After taking a closer look at the working of CSP, we can look at the ways by which we can improve the efficiency of CSP.

a. Improving the operating temperature of CSP. Since CSP works on conventional Rankine cycle, the increase in the temperature, can result in increase in efficiency and convert thermal energy to electrical energy in an efficient manner. It is estimated that, by raising the temperature from 500oC to 700oC can improve the efficiency from 63% to 68% (Carnot Efficiency) [17].

b. Improve the heat capacity of materials at higher temperature, which would in turn result in higher storage capacity. This would reduce the cost of TES system, which would in turn reduce the cost of the CSP plant. It is estimated that by raising the heat capacity by 15% we can reduce the cost of TES by 7% [18].

1.8 Molten salts in CSP

As we have already established that, we can improve the efficiency of CSP by raising the operating temperature from the current 500^{o}C to higher temperature. Commonly used molten salts in CSP are alkali nitrates – Binary salt nitrate (60% KNO3 and 40% NaNO3) and ternary mixture (53% KNO3, 40% $NaNO_2$, and 7% NaNO3). [19].

Table 1.1 Molten salts used in CSP

Sr.NO	Salt	Composition	Decomposition Temp (^{o}C)
1	Solar Salt	60%$NaNO_3$ + 40% KNO_3	450 – 500
2	Hitec Salt	7% $NaNO_3$ + 53%KNO_3+40%$NaNO_2$	450-500
3	Hitech XL salt	48%$Ca(NO_3)_2$+7%$NaNO_3$+45%KNO_3	450-500

Molten salts possess low vapor pressure and better heat transfer properties. But the disadvantages of this salt are high freezing point (around 220^{o}C) as a result of which we need to ensure that the temperatures are maintained and the salt does not freeze. The cost of maintaining the storage material above the freezing point is also a significant one. Another disadvantage of these salts are that they decompose around 500oC. In order to increase the temperature to around 700^{o}C, we need to use other molten salts.

A recent report published by Oak ridge National Laboratory [20], listed out candidates which can be used as secondary coolants for molten salt reactors. The operating temperature of the reactors are in the range of 1000oC. The candidates suggested are Alkali Chlorides and Alkali Fluorides

Table 1.2 Secondary Coolants for Molten salt reactor

Sr.No	Molten Salt	Eutectic Ratio	Melting Point (oC)
1	NaCl-MgCl2	63:37	475
2	KCl-MgCl2	68:32	426
3	LiCl-KCl-MgCl2	9:63:28	402
4	LiCl – KCl	59.5 : 40.5	355
5	LiCl- RbCl	58:42	313
6	LiF-NaF-KF	46.5:11.5:42	454
7	NaF-NaBF4	8:92	384

Since fluorides are toxic in nature, we cannot use at laboratory experiments. So we choose candidate no 4 based on low melting point and cost effectiveness.

The disadvantage of using Alkali chlorides at higher temperature are its corrosion effect and low heat capacity properties. At higher temperature, we use high temperature alloys or ceramic based materials. These materials may also be resistant to corrosion at those temperatures, therefore may not cause any problems.

In order to increase the thermal heat capacity, we disperse nano particles in the eutectic mixtures.

1.9 Nano Eutectics

Nanofluids are stable suspension of nanoparticles in the base liquid. Studies have shown that the thermal properties can be improved by addition of nano particles to base salt [21]. Similar study has been carried out over the enhancement of thermal heat capacity. Shin and Banerjee [22], showed that there is about 31% enhancement in the thermal heat capacity of the carbonate salt with the addition of aluminum oxide nanoparticles.

Table 1.3 Enhancement of Thermal Heat capacity due to addition of nano particles

Sr.No	Authors	Nanoparticle	Base Salt	Concentration	Enhancement
1	Patricia ,Andreu cabedu (2014)	SiO2	Solar Salt	2%	26%
2	Chieruzzi ,Manila et al (2012)	SiO2 + Al2O3	Solar Salt	1%	57%
3	Ho , Ming Xi and Chin Pan (2014)	Al2O3	Hitech	1%	19%
4	Tiznobaik and Shin (2012)	SiO2	Hitech XL	2%	25%

This opens up a range of TES materials with enhanced thermal properties which can also be used a HTF in CSP. Due to this improved properties, we would require small quantity of salt to store the same amount of heat as a result of which the size of supporting structure would also reduce. In this study we try to enhance the thermal heat capacity of eutectic chlorides with addition of nano particles.

1.10 Objective

The purpose of the study is to investigate the effect of nanoparticle addition on a binary chloride mixture and to provide material characterization to understand the anomalous behavior of the salt.

1.11 Significance

We want to reduce the cost of electricity produced by CSP. In order to do that, we are working towards developing new materials which can operate at higher temperature, with better thermal properties and can also be cost efficient. In this study, we are looking at the development of one such molten salt – binary chloride, which can be thermally stable upto 1000°C and when doped with nanoparticles will have better thermal properties. With this enhancement, we can reduce the overall constraints on the plant significance.

Chapter 2

Experimental Procedure

2.1 Experiment Methodology

Based on the previous chapter, we selected the binary chloride mixture of Lithium Chloride and Potassium Chloride. We procured these salts from Spectrum Chemical Manufacturing Ltd. The nanomaterial used was silicon oxide nano particle of 60 NM size. It was also procured from Spectrum Chemical Manufacturing Ltd. All the chemical orders were placed from Fischer Scientific Ltd. In order to make a pure eutectic salt, we measured 88.10 mg of Lithium Chloride and 108.90 mg of Potassium Chloride on a micro scale weighing pan. These weights were calculated based on their molar ratio [b]. These samples were then mixed together in a vial making the total sample weight to 198 mg. similar process is followed to make a nanofluids, where we mix 2% weight of silicon oxide nano particle. This makes the total weight of the nanofluids as 200mg. To ensure that the sample is uniformly mixed it is put through the process of sonication

2.2 Uniform mixing of sample (Sonication)

In order to ensure that the sample is mixed uniformly, we need to mix the sample thoroughly. After mixing the samples in a vial, it manually sonicated with hand rigorously for 2 minutes. This salt is then placed on the hot plate and heated upto 520oC. At this higher temperature, the sample melts uniformly and turns into liquid state. After the melting process, this sample is allowed come down to room temperature. We then scratch the vial for making the sample to prepare the dry sample which can be loaded into the digital scanning calorimeter (DSC).

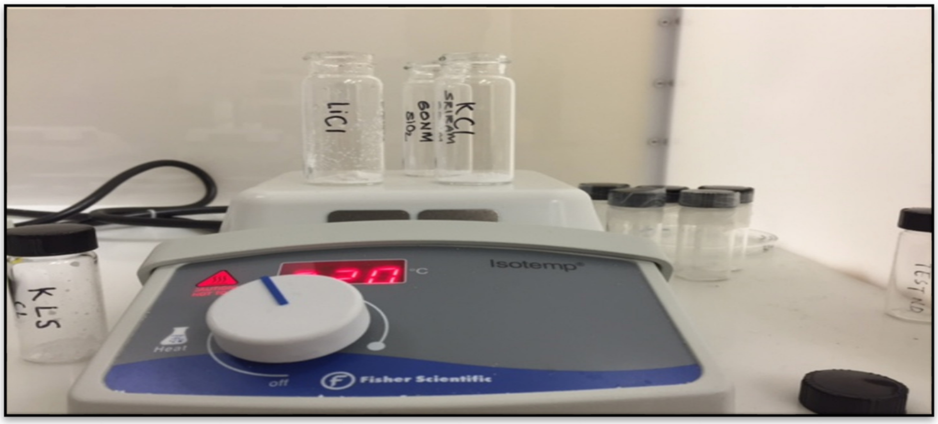


Figure 2.1 Synthesis of Material

2.3 Linear Testing of Sample

We performed the Linear testing of the samples to test the stability of the sample at higher temperature and also to find its decomposition temperature. To perform the linear test we took 10 mg of the sample in a Tzero Aluminum pan and kept an Aluminum lid on top of it. This pan and lid was kept on a hot plate, where the temperature was regulated from 100°C to 500°C at a ramp rate of 50°C/30 mins. At 500°C this sample was kept for 1 hour and later it was cooled down to room temperature. The change in weight of sample was measured. We found negligible difference in the weight of the sample. Thus confirming the stability of the molten salt. This process was repeated twice.

For the next set of test, we hermetically sealed the pan and lid with the sample inside it. Once again it was kept on the hot plate and the temperature was raised to 500°C. After completing the test the change in weight was once again measured and confirmed that the sample did not decompose at 500°C.

2.4 DSC Test (Measure of Heat Capacity)

Digital Scanning Calorimetry (DSC) is used to measure the specific heat capacity of a material. Heat capacity is the amount of heat required to the raise the temperature by 1°C of certain mass of a sample. It is an extensive property of a material. The working principle of the DSC is to maintain a reference sample and the test sample are maintained at the same temperature throughout the experiment. The thermo couple along with a program helps us measure the increment in the temperature along with time and thus the heat capacity is calculated.

Prior to the start of the experiment, the temperature of reference sample was measured and the values were noted. This is the reference graph or the base line graph, with which all the values are compared with. This process is called Calibration of the equipment.

The DSC equipment was procured from TA instruments (QA 20). The prominent feature of the equipment is the provision to use both Modulated DSC and Standard DSC. The difference between them being that, in a MDSC we can vary the ramp rate and ensure that there is a difference in the temperature of the reference and sample to be measured. We can also use it for Heat flow and Latent Heat measurement. In our experiment we used the MDSC.

In order to measure the thermal heat capacity, a portion of the sample was scratched and loaded on a Tzero Aluminum pan. We need to ascertain that, no sample is loaded on the shoulder of the pan to avoid contamination of the equipment. We then kept the sample on the hot plate at 520°C for 5 minutes, to allow the sample to melt completely and form a uniform shape. After the melting process is completed and sample is in the powder form, we seal the pan with the lid (Tzero Aluminum Lid) and then seal it hermetically. This sealed sample along with reference sample is loaded in the DSC on the handle. We set the protocol for measurement by allowing the DSC to start from 40^{o}C and ramp at 5oC/min till 500^{o}C. Then at 500oC it is allowed to be at isothermal conditions for 1 minute. After this, the sample is allowed to cool till 40oC. All the data is recorded in the DSC, and we generate a Heat Capacity (Cp) vs Temperature (T) graph.

Similarly, when we prepare the sample with silicon oxide nanoparticles and we carry out the same procedure to measure the heat capacity and the heat capacity vs Temperature graph is generated. We can see that there is enhancement in thermal heat capacity. In order to ensure the repeatability of the test, we repeated the test thrice and saw consistency in the results.

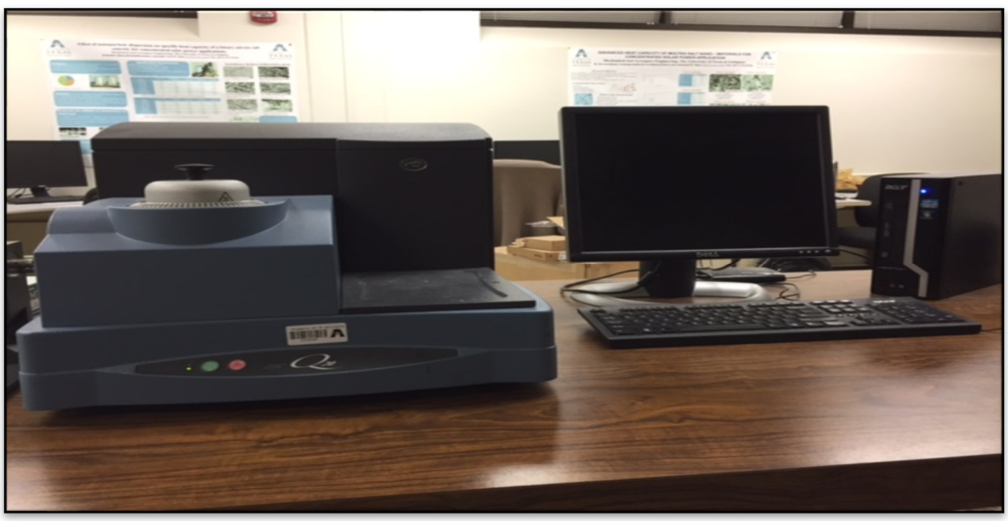


Figure 2.2 Modulated Digital Scanning Electrometry

2.5 Material Characterization

Material characterization is the method in which we study internal structures of the materials using external techniques. It helps us to study the molecular structures along with interaction of intermolecular forces. The material characterization techniques used in this study are Scanning Electron Microscope and Energy Diffusive X –ray Spectrometry (EDS)

The working principle of Scanning Electron Microscope (SEM) uses high velocity electrons bombarding the sample in vacuum. This electron colliding with the material produces signals, which helps us understand the intermolecular bonding. It generates a 2 dimensional image, which helps us understands the structure better. The SEM used in this study was HITACHI.

Figure 2.3 Scanning Electron Microscope

Similar to this is EDS analysis, EDS stands for Energy Diffusive X –ray spectrometry (EDS) which is used for the fundamental analysis or chemical characterization of a sample. To generate X – rays high energy beam is focused into the sample creating an electron hole pair and thus emitting X rays . The EDS system used in this study was Thermo Fischer.

We tested the pure sample initially and then with nano materials to understand the change in the internal structure due to the addition of nano material and to substantiate the DSC results.

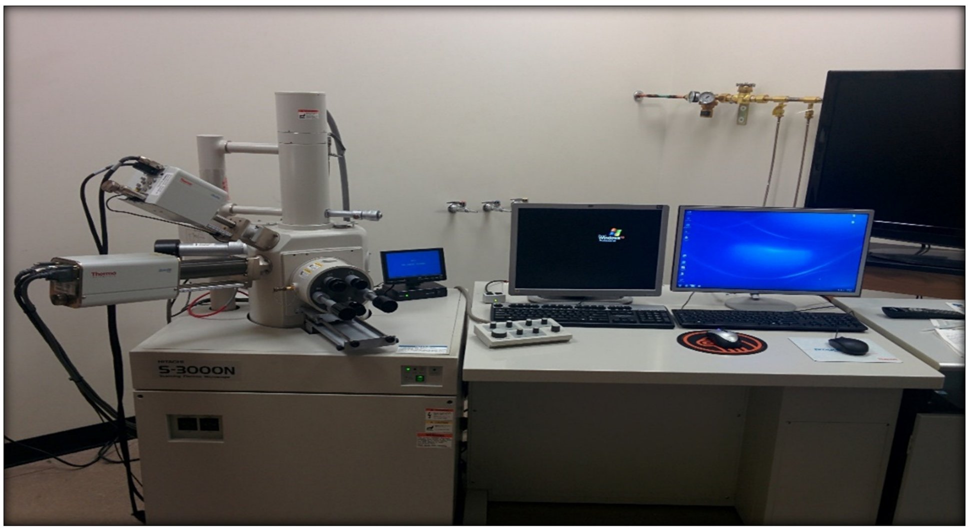


Figure 2.4 Energy Diffusive Spectrometry

Chapter 3

Results and Discussion

3.1 MDSC results

We carried out the heat capacity measurement of binary chloride sample along with the nanoparticles sample. We prepared the pure samples initially and tested them in the MDSC and the results were recorded. The heat capacity value was found out to be 1.34 – 1.36 KJ/Kg^{o}C , which is in agreement with the literature value [15]. To confirm these results, we tested the samples thrice. Thus we observed the repeatability.

We later added nanoparticles 60NM (2% weight) to the pure sample and tested them in the MDSC and recorded the results. There was an enhancement in the value of heat capacity. The values recorded were 1.55-1.57 KJ/Kg^{o}C. We repeated the experiments thrice to confirm the results.

The pure samples were tested and the average value was measured to be 1.31 KJ/Kg^{o}C. This is in accordance with out literature value (1.30 ~1.33 KJ/Kg^{o}C). Then we tested the nano eutectic samples, and the enhancement of thermal heat capacity was measured. We observed around 15% enhancement in the liquid phase and 12% enhancement in the solid phase. The combined results along with graphs is shown below.

Table 3.1 Enhancement of Thermal Heat capacity – Liquid Phase

Sr.No	Heat Capacity (KJ/KgoC)	Pure Sample	Nano Eutectic
1	Test #1	1.28	1.50
2	Test # 2	1.31	1.52
3	Test # 3	1.33	1.53
4	Average	1.31	1.51
5	Enhancement		15%
6	Experimental Uncertainty	2.08%	1.2%

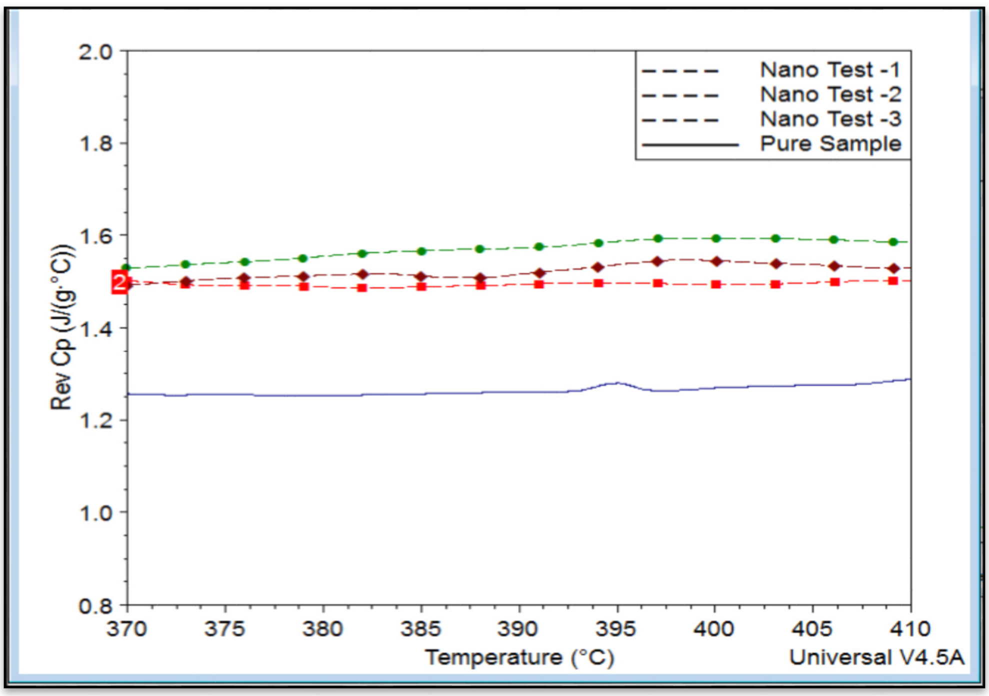


Figure 3.1 Pure eutectic vs Nano Eutectic (Liquid Phase)

Table 3.2 Enhancement of Thermal Heat capacity – Solid Phase

Sr.No	Heat Capacity (KJ/KgoC)	Pure Sample	Nano Eutectic
1	Test #1	0.86	0.93
2	Test # 2	0.92	1.01
3	Test # 3	0.87	1.04
4	Average	0.88	0.99
5	Enhancement		12%
6	Experimental Uncertainty	2.6%	4.6%

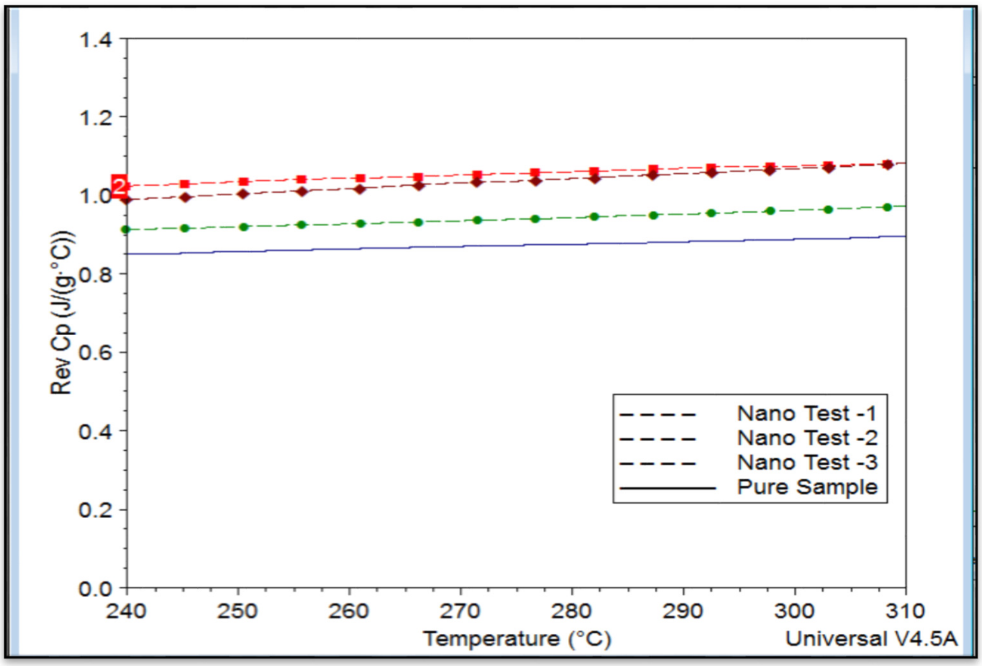


Figure 3.2 Pure Eutectic vs Nano Eutectic (Solid Phase)

3.2 Scanning Electron Microscopy

We obtain the pure image and nano eutectic image under a scanning electron microscope (SEM). We observe change in molecular structures which are observed in the images. Let us take a close look at the images.

Figure 3.3 Pure sample image without the presence of nano structure

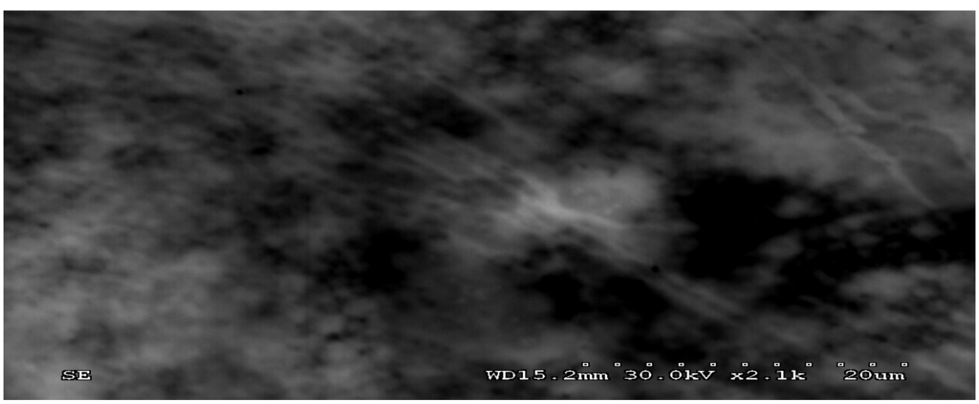


Figure 3.4 Image of Nano Eutectic with presence of Nano structures

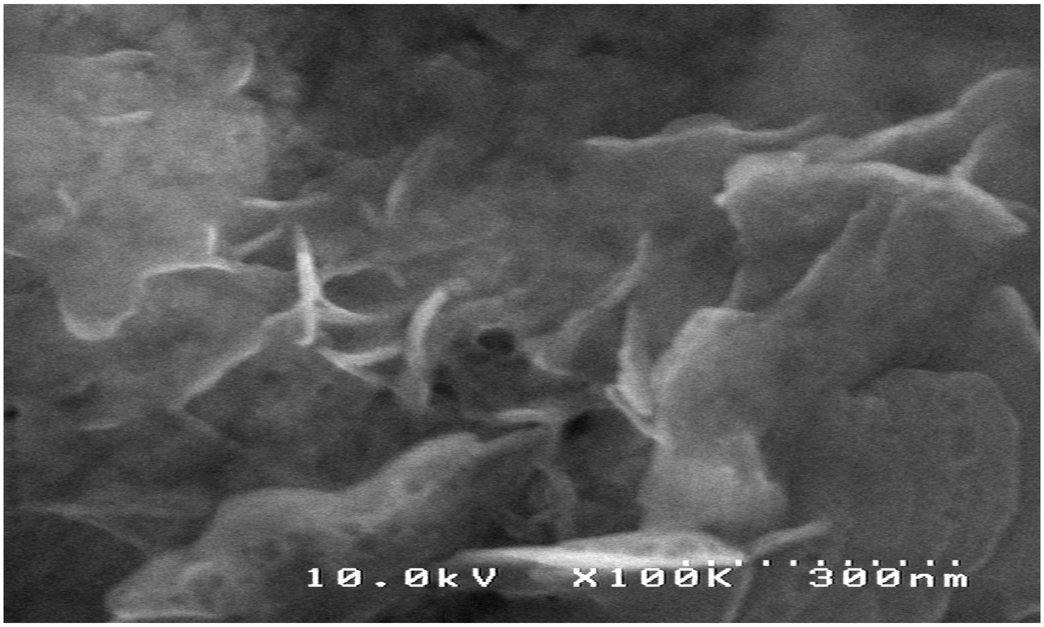
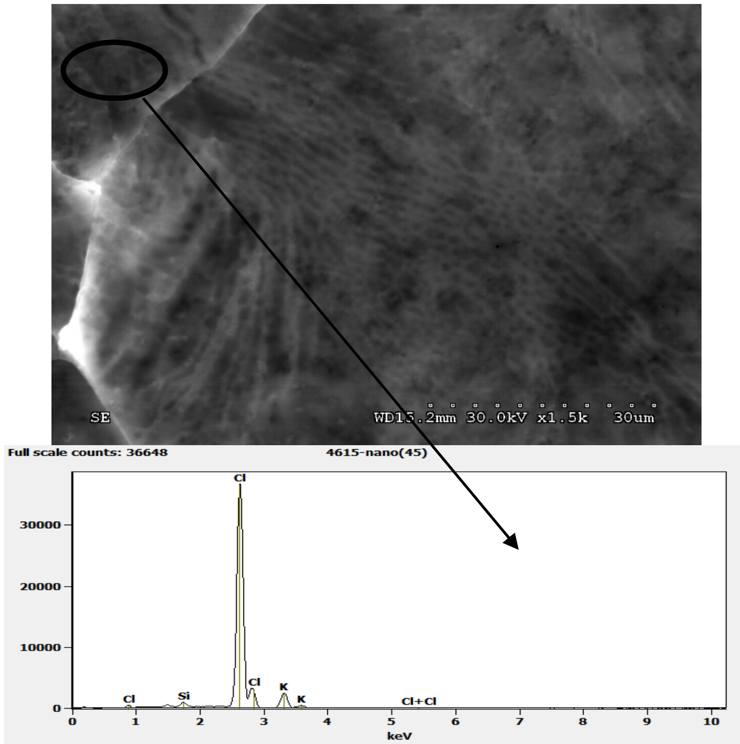


Figure 3.5 High definition image of Nano structures in Nano Eutectic

We observe these needle like nano structures formed in the nano eutectic sample. These structures have higher surface area as compared to the bulk area. It is because of these structures that the specific heat capacity of the sample increases.

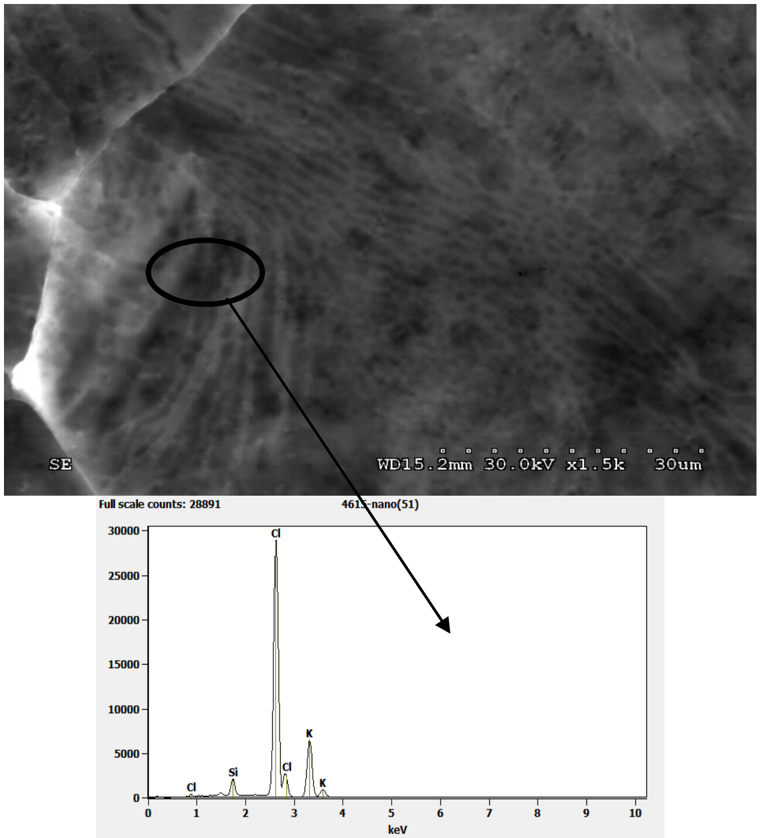
3.3 Energy Diffusive Spectrometry (EDS)

In order to understand the nanostructures formed, we perform the EDS analysis over the nano eutectic sample.



Element Line	Weight %	Weight % Error	Norm. Wt.%	Norm. Wt.% Err	Atom %	Atom % Error
Si K	1.09	± 0.04	1.09	± 0.04	1.39	± 0.06
Cl K	85.71	± 0.25	85.71	± 0.25	86.52	± 0.25
K K	13.20	± 0.14	13.20	± 0.14	12.08	± 0.13
Total	100.00		100.00		100.00	

Figure 3.6 EDS analysis of Bulk area



Element Line	Weight %	Weight % Error	Norm. Wt.%	Norm. Wt.% Err	Atom %	Atom % Error
Si K	3.40	± 0.05	3.40	± 0.05	4.37	± 0.07
Cl K	66.96	± 0.21	66.96	± 0.21	68.24	± 0.22
K K	29.64	± 0.18	29.64	± 0.18	27.39	± 0.17
Total	100.00		100.00		100.00	

Figure 3.7 EDS analysis of Nanostructures

Comparing the EDS analysis of the bulk area and Nanostructure area we can conclude that:

1. The Nano structures are formed only in few areas as compared to the entire salt.
2. The nano structures are formed due to the interaction of potassium molecules with silica nano particles.

3. These nano structures have high surface area, which results in increase in thermal heat capacity as compared to base sample.

Chapter 4

Conclusion and Future work

4.1 Conclusion

We observed the effect of nanoparticle addition on a binary eutectic chloride mixture. This binary mixture of Lithium and Potassium chloride was doped with 2% weight of silica nano particles and the enhancement in thermal heat capacity was recorded. We saw there was a 15% enhancement in liquid phase and 12% enhancement in solid phase. These samples were further observed under a scanning electron microscope to observe the nano particle behavior. We saw that needle like structures called Nano structures are formed due to addition of nano particle in the base salt. These nano structures have enlarged surface area as compared to the bulk material, which results in the enhancement of thermal heat capacity. The nano structures were further studied using Energy Diffusive Spectrometry. With the analysis, we concluded that these structures are formed due to the interaction of K^+ ions with silica nano particles.

This eutectic chloride with enhanced heat capacity can be used TES and HTF material at higher temperature (around 700^oC). Due to this, we can increase the operating temperature from 500oC to 700^oC, thus improving the Carnot efficiency of the system. With the enhanced heat capacity, we can store more energy at higher temperature for the same quantity of salt, thus we can reduce the size of TES system.

4.2 Future work

The future work for the alkali chloride mixture can be as following:

1. Checking the Thermal and Chemical Stability at higher temperatures.
2. Viscosity measurement.

3. Checking the effect of additional alkali chlorides with the base salt like ternary or quaternary chloride mixtures.
4. Checking the effect of different nano particles like Aluminum oxide and Titanium oxide particles.

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Biographical Information

Sriram Sambasivam was the youngest son on M Sambasivam and S Bavani. He completed his Bachelors of Mechanical Engineering from Mumbai University. After completing his Engineering , he worked with VOLTAS Ltd (a TATA Enterprise) as a Senior Engineer – Execution for 2.7 years after which he started his Masters in Mechanical Engineering from University of Texas at Arlington.