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Slats Hydrates -Solar Heat Energy Storage

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Abstract—

In this paper, the slats hydrates of CaCl2 and Vermiculite were tested as possible thermo-chemical heat storage materials for seasonal heat storage. These two materials were mixed to form one salt crystal sample and since two batches of Vermiculite were used then two crystal samples formed and tested. The carried out tests were TGA and DSC tests in which the loss in mass and heat flow were measured as temperatures increases from 25 to 150 degree. The energy analyses showed that two endothermic reactions occurred one at temperature of 29 C and the other at temperature of 45 C for the two samples. The reactions appeared as peak areas at the generated plots. Fully reversible reactions were governed the storage and release of thermal energy through the dehydration and hydration of chemical salts. At 45 C a mass loss of 3% was occurred. As a result, it was found that combining chemical salts within porous host matrices or what known as (Salt in matrix – SIM) has revealed to enhance the reaction rate of the chemical salts and reducing hysteresis throughout the charge and discharge cycles in addition to avoiding the deliquescence of the salt throughout the hydration phase.

Keywords—Solar enery storage, sotrage materials, hydration and dehydartion, TGA and DSC analysis, vessel design.

I. INTRODUCTION

Throughout winter, high amounts of energy is being utilized to produce heat for domestic requirements like space heating and water heating which are produced from the process of burning non-eco-friendly materials such as, fossil fuels, etc. In an alternative way, the needed energy can be produced through using an eco-friendly and economical solar energy storage technology with few amount of pollution. Throughout summer, surface of the earth receives adequate sunshine which is used to produce the needed heat energy for winter season [1]. Depending on fossil fuel to generate energy leads to generate huge amount of carbon emissions. This research introduces new approach to generate chemical heat energy using dehydration reactions. It is expected to minimize the carbon footprint and global warming. The chemical reaction heat can be used to heat air and water for domestic uses.

Several candidate systems are available and currently applied to deliver domestic thermal energy storage (TES) like phase change materials (PCM), highly insulated building envelopes coupled with thermal mass, tanks of hot water and electrical storage heaters. All of these systems are mainly planned to only deal with diurnal energy demand. Conventionally, water was utilized for solar heat storage (such as solar boiler) for short periods of time. When long term heat storage is needed, then this needs a large water tank more than 25-50 m^3 which usually very large and needs a large space to place it inside a building [2] . Lately, an alternative choice for inter-seasonal thermal energy storage has emerged utilizing salts entrapped within porous desiccant materials (i.e. Salt In Matrix—SIM). These systems use reversible thermo chemical reactions in order to store solar energy effectively which may later be released as heat. This method is usually known as thermo-chemical materials (TCM). These materials are known with their theoretical energy large densities compare to water. The basic reaction process for thermal energy storage using TCM is [3];

$$\text{C (solid +Q (heat)} \longleftrightarrow \text{S (Liquid/gas) +B (Solid)}$$

Throughout summer, the thermo-chemical material (known as Solid C) divides into the gas or liquid (A) in addition to the solid B due to the heat addition from a solar collector. For materials A and b, they can be separately stored until a later time. On the other hand, the materials A and B are mixed in winter in order to begin the reverse reaction to release the solar heat that was stored. Since the two materials are separately stored, then no reactions take place which means that thermo-chemical storage can be utilized for heat transportation and loss-free storage. In general, TCM materials are featured with non-corrosive nature, non-toxic, cheap, have enough energy storage density as well as having reaction temperatures in the correct range. These necessities are satisfied through lots of salt hydrates by means of this reaction [3];

$$\text{Salt.n}H_2O\text{ (s) +heat} \longleftrightarrow \text{Slat (s) +n}H_2O\text{ (g)}$$

The most promising salt hydrates materials for seasonal heat storage are; ($CaCl_2.2H_2O$), ($MgCl_2.6H_2O$), ($Al_2SO_4.18H_2O$) and ($MgSO_4.7H_2O$). [4], utilizes MgSO4 as seasonal heat storage material. The atmospheric pressure in the experimental work indicates that this material is capable to obtain and release nearly 10 times (by weight) more energy compared to water storage [4]. Their results also presented that the hydration of MgSO4 proceeds quite slowly (5-20 hours) which limits a fast and full release of the stored solar heat, especially for a thick layer of material. The potential of the listed above four materials as TCM material for heat storage was usually examined under low pressure circumstances and this for inert gases to removal that found in the system in addition for pressure drop reduction among water and salt [4].

II. Literature Review

A. Heat storage materials

Several materials can be used in thermal energy storage systems depend on the type of the system. According to the heat storage mechanism, these materials can be classifies as; chemical reaction, absorption, adsorption, latent and sensible. The following figure presents a small materials range appropriate for TES applications. As shown in the figure and by reading from left to right, the mechanism of thermal energy storage causes an increase in the energy density, Ed or what called storage potential which refers to the quantity of accumulated energy per unit volume or mass [5].

Materials like building materials rock and are sensible storage materials. In this type of materials, the thermal energy storage occurs as a result of a temperature rise of the material. These material are known with their low Ed which is given as the product of the material specific heat by any temperature change [6]. On the other hand, PCM's have higher Ed which lead to increased TES ability. For the thermo-chemical reactions materials, they provide the highest Ed among all types of materials groups [7]. To characterize the chemical reactions between compounds, the variation in the molecular configuration of these compounds will be applied [8]. Any inter-seasonal TES system may deeply take advantage from having materials that have a a very high Ed thus decreasing storage volumes and/or mass in addition to offering enough output in term of heat energy.

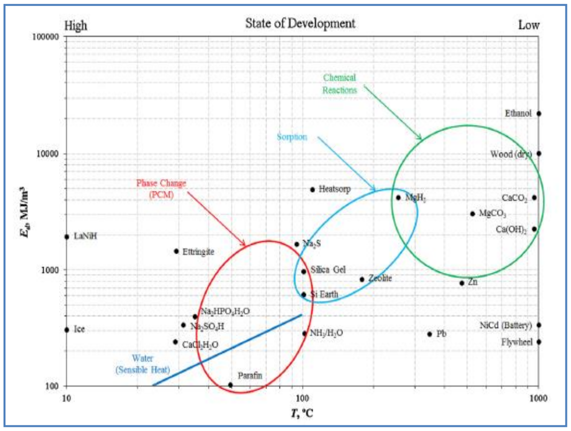

Figure 1: Energy densities of different classes of materials Adaptedfrom [9].

Different types of thermo-chemical materials can be effectively used for TES purposes. These materials involve Calcium Chloride ($CaCl_2$), Lithium Bromide (LiBr), Lithium Nitrate ($LiNO_3$), Magnesium Sulphate ($MgSO_4$), Calcium Nitrate($Ca(NO_3)_2$), synthetic zeolites as host matrices, silica gel, vermiculite and activated carbon. This project interested on testing two types of salt materials which are Calcium chloride $CaCl_2$ and vermiculite [9].

B. Hydration and dehydration of salt materials

Salt hydrates is deemed as same as inorganic salts alloys and water, producing an ideal crystalline form. Their phase alteration transmission can essentially be observed as either a hydration or dehydration of the salt, even though that operation is as same as the freezing or melting the composite. In general, salts hydrate typically melt to salt hydrates together with smaller water moles' number or their anhydrous formula. Pending the phase conversion transmission, the hydrated salts dissolve in the water, which is produced of the created non-hydrated salt particles. It is good to mention that the salt hydrates are considered attractive for heat accumulating aims in residences since their volumetric storage density is great and thermal conductivity is quite great too matched to organic phase change materials. In addition to that, salt hydrates are considered lower cost. On the other hand, a main trouble with salt hydrates might be created of the reduction of the energy storing density with cycling [10]. One of that trouble's reasons is the incompatible melting. This trouble was defined as: pending the salt hydrates' melting, the flowed water is considered not adequate to dissolve the whole existing crystals. Therefore, as a result of their density, these crystals deposit at the bed of the vessel as well as will not recreate a hydrated crystal form pending the following solidification stage of the composite, which leads to reversibility in the operation [11].

In general, Dehydration reactions include monomer molecules such as simple sugars or amino acids which are bonding together. As they are bonded, both of OH and H (H2O) are eliminated from them so that its known as dehydration since the bonded molecule loses water. The below figure demonstrates the dehydration reaction. In the figure, the blue-shaded circles related to individual monomers [12].

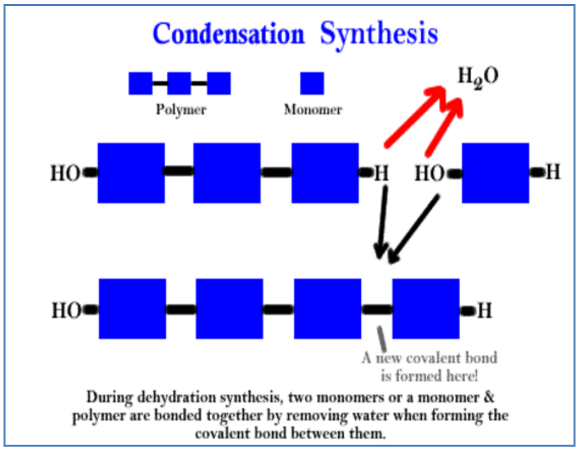

Figure 2: Dehydration reactions [12].

The lattice crystallization of calcium chloride is favored in this topic because the lattice energy of crystallization is positive which indicates that it is endothermic reaction. The lattice constant of this material are: a = 6.2Å, b = 6.444 Å, and c = 4.17 Å.

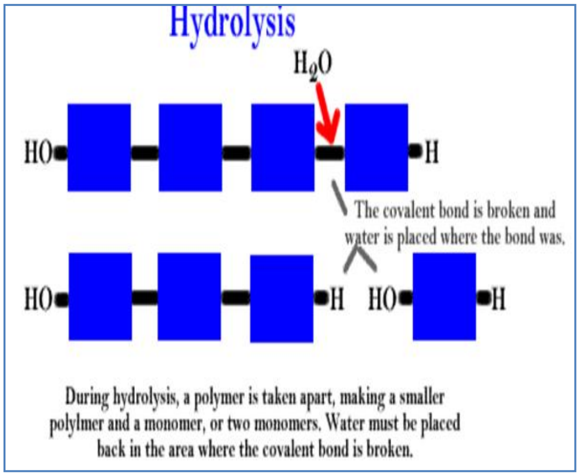

Figure 3: Hydration reaction [12].

From the above figures, it can be concluded that the only difference between the two processes is that in of them bonds are being formed by water removal, while in the other bonds are being destroyed by water addition to dissolve bonds. Also, it can be conclude that Dehydration Reactions energy is added o organic molecules while in Hydrolysis Reactions energy releases from the molecules.

III. Experimental Methods

A. Simultaneous thermal analysis (STA)

The hydration and dehydration processes can be thermally analysed through applying what called STA (simultaneous thermal analysis). This type of analyses is mainly divided into Thermo gravimetric analysis (TGA) and differential scanning calorimetry (DSC) [13]. Both TGA and DSC approaches were followed in this work to achieve integrated method of them called STA.

i. TGA Analysis

Thermo gravimetric analysis is used to measure the change in mass with temperature change. The following schematic diagram represents the TGA measurement principle. The placed sample on this device is exposed to a heat source and heated under synthetic air or nitrogen with constant heat rate. The change in the mass throughout this process is then measured or what called mass loss. This measurement is an indication that a degradation of the measured substance occurs. In case of applying oxygen from synthetic air, the reaction may result to an increase of mass.

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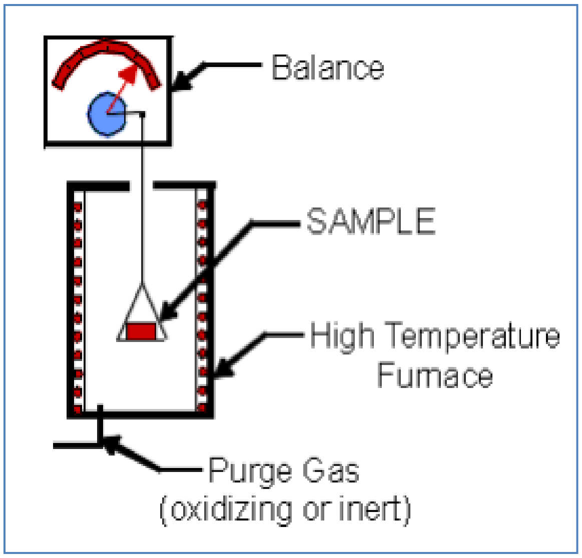

Figure 4: Schematic principle of TGA measurement [13]

ii. DSC device;

The DSC device is utilized to measure the change in heat flow as temperature changes. It can be utilized to measure the thermal critical points such as glass transition temperature, specific heat, enthalpy or melting point. The following schematic diagram shows the principle of the DSC device. In this device, both of the sample and an empty reference crucible is heated at constant heat flow. A change in the temperature of both crucibles resulted via the sample's thermal critical points can be then detected.

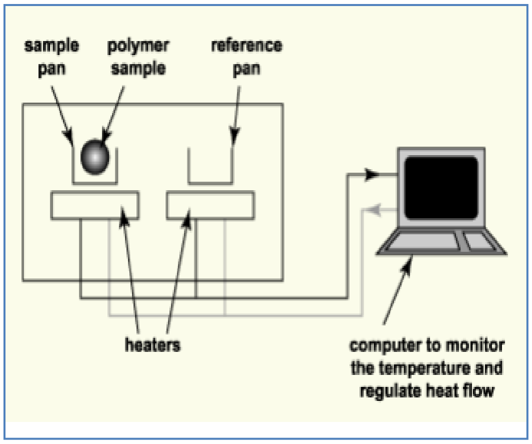

Figure 5: Schematic principle of DSC measurement [13]

Both TGA and DSC approaches were followed in this work to to analyze changes in sample mass and heat fluxes throughout hydration and dehydration reactions. Also, solar heat storing vessel which can trap heat energy in summer and move it inter-seasonally to winter and can play an important role in reducing the amount of carbon generated from fossils will be designed. The design will consist of a glazed steel collector which will get hotter than the actual atmospheric temperature. This heat energy will then be transferred into a storage vessel containing salt carrying materials which have high thermal capacity like $CaCl_2$ (calcium chloride). This is achieved by using chemical reactions by the hydration and dehydration process of inorganic salts. As the salt and water are held separately, this means that there will be no energy dissipation during storage. Once the salts are hydrated, the heat energy stored will get released and can be used for several months. Different heat transferring systems/networks will be examined and compared for energy and economic performance. A choice of stainless and plastic pipes will be used to transfer heat. Stainless steel is less corrosive than plastic but on the other hand, plastic requires less maintenance.

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- **Experimental tests**

A series of experimental tests were carried out on two different types of salts which are CaCl 2 (calcium chloride) And Vermiculite as solar thermal energy storage materials. The experimental tests were divided into two parts, where in the first part the two salts were mixed and their masses after and before mixing were measured while in the second test an simultaneous thermal analysis was conducted. Appendix shows the tested system that was designed by the researcher.

B. Salts mixing

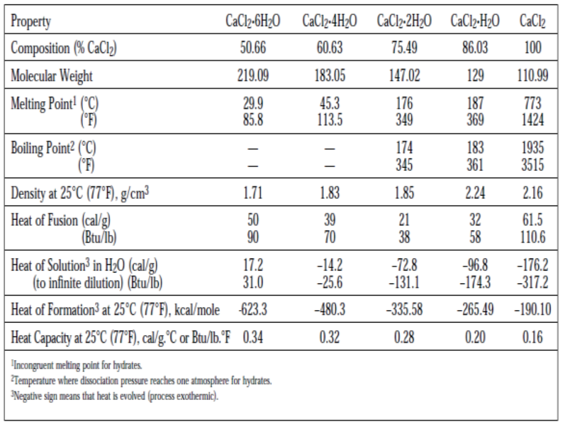
The overall test based on adding Cacl2 on the vermiculite slat so that salt crystals were formed on the vermiculite. The two mixing materials have the following properties;

- **Calcium chloride**

Calcium chloride is considered as one of the major versatile of the fundamental chemicals. it was knows and used for more than 100 years. Its characteristics and properties make it a good choice for extensive range of applications. The available data on literatures regard its physical properties, its hydrates and solutions are generally denotes to pure material. It has four main commercial grades knows as the LIQUIDOW, DOWFLAKE, PELADOW and PELADOW DG brand calcium chloride. They include additional trace elements and compounds that are fabricated within the defined limits by The American Society for Testing and Materials (ASTM) Standards. Table below illustrates the key Physical Properties of Calcium Chloride Hydrate [14].

At ordinary temperatures, solid phase separation will takes place under specific temperature concentration circumstances although calcium chloride is highly soluble material in water.

Table 1: Physical properties of CaCl2 hydrate[14]



Property	$CaCl_2 \cdot 6H_2O$	$CaCl_2 \cdot 4H_2O$	$CaCl_2 \cdot 2H_2O$	$CaCl_2 \cdot H_2O$	$CaCl_2$
Composition (% $CaCl_2$)	50.66	60.63	75.49	86.03	100
Molecular Weight	219.09	183.05	147.02	129	110.99
Melting Point[1] (°C) (°F)	29.9 85.8	45.3 113.5	176 349	187 369	773 1424
Boiling Point[2] (°C) (°F)	— —	— —	174 345	183 361	1935 3515
Density at 25°C (77°F), g/cm^3	1.71	1.83	1.85	2.24	2.16
Heat of Fusion (cal/g) (Btu/lb)	50 90	39 70	21 38	32 58	61.5 110.6
Heat of Solution[3] in H_2O (cal/g) (to infinite dilution) (Btu/lb)	17.2 31.0	-14.2 -25.6	-72.8 -131.1	-96.8 -174.3	-176.2 -317.2
Heat of Formation[3] at 25°C (77°F), kcal/mole	-623.3	-480.3	-335.58	-265.49	-190.10
Heat Capacity at 25°C (77°F), cal/g.°C or Btu/lb.°F	0.34	0.32	0.28	0.20	0.16

[1] Incongruent melting point for hydrates.
[2] Temperature where dissociation pressure reaches one atmosphere for hydrates.
[3] Negative sign means that heat is evolved (process exothermic).

These circumstances are illustrated by the phase diagram of the CaCl2-water system presented in the figure below.

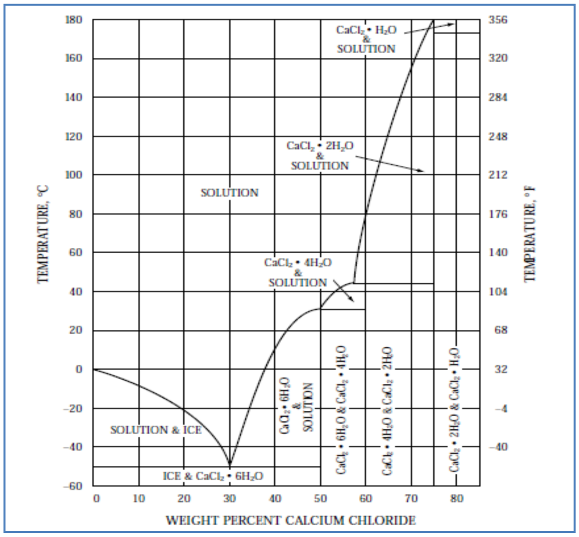


Figure 6: Phase diagram for CaCl2 and water solutions [14].

- **Vermiculite**

For the second material, it is group of hydrated aluminium/ iron/magnesium silicates with a laminate

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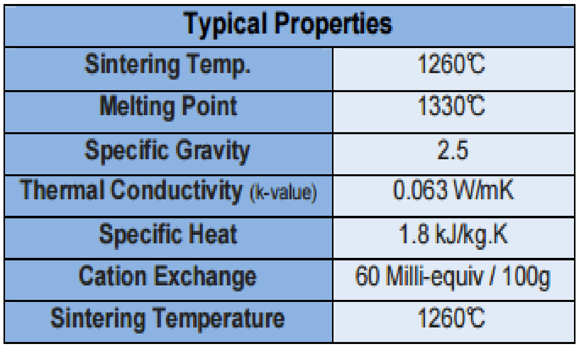
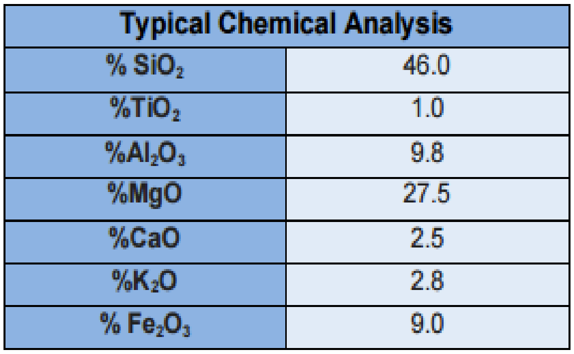
structure that are naturally occur. It has a proposed formula of $(MgFe+2Al)_3(Al,Si)_4O_{10}(OH)_2\cdot 4H2O$). For this material, the raw material is pulverized and is then exposed to direct heat inside a furnace. As a results of this heating process, it 'exfoliates' or expands in size, into a form comprising of a group of parallel wafers with air spaces among them. for the exfoliated vermiculite, it is created as a granular loose fill which may be bonded to make dry mixed or boards with fillers and binders for spray application. for CRUDE VERMICULITE, it involves flat and thin flakes comprising microscopic layers of water. The estimated maximum operating temperature of vermiculite is 1100°C. It has a color ranges from black and different shades of brown to yellow for the flakes, varies at expansion (exfoliation) to bronze or gold. The consequence lightweight materials from raw flakes expansion are characterized with their odorless, fire resistant and chemically inert properties. When it is used in lightweight concrete or plaster, vermiculite delivers good thermal insulation. It has the ability to absorb liquids which can then be transported as free-flowing solids. Tables below show the physical and chemical properties of vermiculite [15].

Table 2: physical properties of vermiculite [15]



Typical Properties	
Sintering Temp.	1260℃
Melting Point	1330℃
Specific Gravity	2.5
Thermal Conductivity (k-value)	0.063 W/mK
Specific Heat	1.8 kJ/kg.K
Cation Exchange	60 Milli-equiv / 100g
Sintering Temperature	1260℃

Table 3: Chemical properties of vermiculite [15]



Typical Chemical Analysis	
% SiO_2	46.0
%TiO_2	1.0
%Al_2O_3	9.8
%MgO	27.5
%CaO	2.5
%K_2O	2.8
% Fe_2O_3	9.0

The followed experimental procedure for the hydration and dehydration of these two materials was a followed;

- A balance was used to weigh two batches of vermiculite which were 300g and 310g.
- A 3.5 ml of Cacl2 solution was then added to every 1 gram of vermiculite and then the vermiculite with the solution were mixed properly. This amount of calcium chloride that was added constitutes the ultimate amount required to get enough salts formed on the vermiculite.
- The mixed salts were then left to dry for 1 hour to absorb moisture and then bake them in the oven for 24-48 hours in order to remove moisture in the salts.

IV. Results

The results of this part are provided in the following table;

Table 4: measured weights of two materials

Salt Type	Batch #	Before	After	Salt (g)	Final Salt (g)
		V (g)	V+Salt (g)		
$CaCl_2$	1	300	900	600	600

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	2	310	950	640	640

The ratio between the mass of calcium chloride salt to the mass of Vermiculite was measured as shown in the following table for the two batches;

Table 5: Average ratio of masses

Batch #	**Ratio of gs/gv**	
For batch 1	2.00	**Average ratio is 2.03**
For batch 2	2.06	

STA (simultaneous thermal analysis) test

In this test, measurements for mass change and heat flow were done. It is a combination of: TGA (thermo gravimetric analysis) which measures the change of mass with respect to temperature and DSC (differential scanning calorimetry) which measures energy changes with respect to temperature. For this test, the prepared small crystals from the two batches in the previous part were used to carry out the STA test. The followed procedure for the STA test was as follows;

- The sample of the salt crystal was placed on the machine at where it was weighed.
- The sample was then removed and humidified up to a certain weight using a humidifier and then weighed again in the STA machine.
- The STA test was then run through applying air through the machine to start the test.

As a result, it was found that the dry weight of the first sample was 22.95mg while the dry weight of the second sample was 22.4mg. The humidification required for the crystal was calculated and the results of calculations are presented in table below;

Table 6: Humidification results

Salt	**CaCl2**
SIMs (g)	1
gs/gy	2.03
Matrix	0.329787
Salt	0.670213
# of moles	0.006038
H2O molecules	4
Molar mass H2O	72
Mass increase (g)	0.434733
Final SIM Mass	1.434733

The above table shows that; the final weight of the first sample after humidification was 32.971mg and the weight of the second sample after humidification was 32.138mg. It must be noted that the calculation were made based on the following molar masses of the reaction components.

Table 7: molar masses of reaction components

Chemical/Compound	**Molar Mass (g)**
H	1
O	16
Ca	40
Li	7

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Mg	24
Cl	35.5
N	14
S	32
H2O	18
CaCl2	111
LiNO3	69
MgSO4	120

The obtained results from the TGA and DSC tests on the materials are presented in a plot format that show the relation between the mass loss and heat flow and the temperature for the two materials.

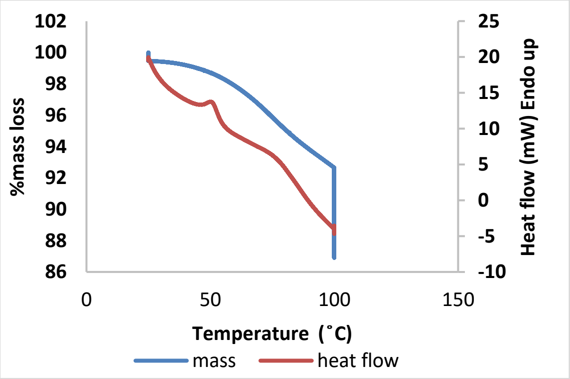

Figure 7: Hydration and dehydration results of the first salt crystal sample as a function of temperature.

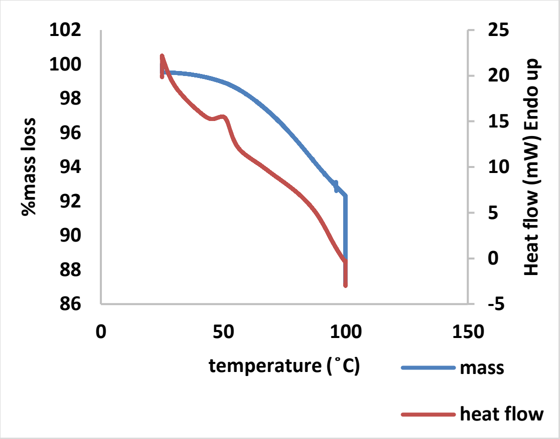

Figure 8: Hydration and dehydration results of the second salt crystal sample as a function of temperature.

For the energy analysis, the reaction is positive endothermic reaction. The CaCl2 has two dehydration stages within the current experimental setup:

- $CaCl_2$. 6H2O à $CaCl_2$. $4H_2O$ @ 29.9Deg C (Dow corning reference)
- $CaCl_2$. $4H_2O$ à $CaCl_2$. $2H_2O$ @ 45.3Deg C

The rig was tested and the following results were obtained.

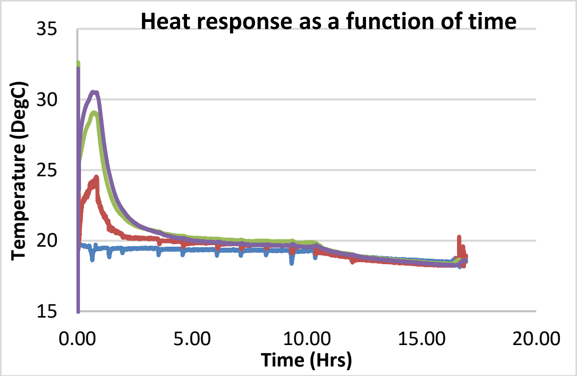

Figure 9: Temperature profile of the rig for different cases.

V. Discussion

The experimental works showed that thermo-chemical storage through the hydration/dehydration reactions of chemical salts delivers a proficient ways of energy storage for long term. This occurs because high energy densities can be potentially obtained and because the carried out reaction is completely reversible and this clear with the given below equation

$$\text{Salt.x(H2O) + heat} \longleftrightarrow \text{Salt +x(H2O)}$$

Where x refers to the number the hydrated states.

The measurements were conducted with around 32.799 mg of material weight. These measurements were used to analyze changes in sample mass and heat fluxes throughout hydration and dehydration reactions. The mixture humidity was measured at the oven outlet and utilized for calculation of the water vapour partial pressure within the device oven. The utilized temperature program for samples dehydration before the hydration was set to ramp up with 5.0ºC/min. some actions were done immediately such as; Switching the Gas to Air at 20.0 ml/min, Holding for 10.0 min at 25.00°C, Heating from 25.00°C to 100.00°C at 5.00°C/min and then Holding for 10.0 min at 100.00°C.

The TGA element represented as a change in the gradient of the curve and the DSC element showed as a positive bump and this because the endothermic reaction is positive in this setup.

The obtained results in Figures 7 and 8 for the mass loss and heat flow against temperature increase showed the dehydration results of the two salt crystals samples after applying the TGA and DSC tests on them. In both cases the data is fairly consistent.

Also, in both figures, two clear peaks and gradient changes are resulted. These results showed that at the beginning of the experimental analysis, there is a major mass drop on the TGA results and an endothermic peak straight away. This occurs at the temperature value between 26-30 °C. At this point, the reaction is presented in the following figure where no loss in the mass occurred till now.

A second bump occurs at around 50 °C, again with an associated gradient change. These are representative of the second reaction in which the loss of mass was 97% as shown in the following figure which means 3% of mass was lost.

Interestingly, a third smaller bump also appears around 70 °C and this probably occurred due to a few reasons such as; the transportation of moisture from the internal region of the SIM to the external region and then removal from the salt all together, or due to a delay in warming the central region of the SIM and that the moisture is removed at a later time or due to that the thickness of the salt is differing and it takes longer for the heat to penetrate the thicker layers.

By comparing the obtained data in table 1 for the properties of calcium chloride with those data obtained experimentally, it was found that they are very close since at the first amount of compounds of $CaCl_2.6H_2O$, the applied heat to SIM forces the dehydration reaction 17.2 cal/g as given in the table and this value is very close to that obtained in the experimental results. These close readings validated the calculations done for the energy transfer from SIM to water.

The obtained results showed that a closed low pressure heat storage system is needed to achieve a successful implementation of salt hydrates and this will be done in the next stages where a vessel with different distribution system will be analyzed. The following schematic diagrams showed the overall diagram of the storage vessel, hydration reaction layout, dehydration reaction layout and the fourth suggestions of the distribution systems.

As shown in Figure 9, four thermocouples were installed at different locations to measure the temperature of air around the rig. It can be noted that the maximum temperature can be obtained is only 31°C while the optimal design should reach 40°C. The reason behind this result is the initial design calculations and the assumptions were taken during the mass calculation of the salt mixture.

VI. Conclusion

The energy generation is very important for different industrial application and urban usages. Most energy is generated in the world depends on fossil fuel which leads into many environmental impacts and issues such as; global warming and CO_2 emissions. In the other hand, there are many sources of clean and renewable energy can be used to heat or cool different systems. In this research, the chemical heat of hydration and dehydration reactions was studied to be used in heating domestic air and water. The idea is to promote and control the hydration and dehydration of calcium chloride where the released heat during the reaction will be used to heat the ambient air inside a control volume or it can be used for water heating in the winter. The methodology of the research includes design and synthesizes the material and testing the ability of the system to heat air blowing over the reaction bed. It is worth mentioning that a process flow diagram of the rig was designed and the bed was sized based on chemical calculations. Using calcium chloride and vermiculite, a new solid material was synthesized and tested based on chemical calculations. The synthesized material was packed in a tested rig and both TGA and DSC approaches were followed to test the performance of new salt combination. The final testing show that the maximum air temperature can be achieved is 31°C using the synthesized salt. It is worth mentioning that the design constrain is to heat the air into 40°C or higher. This constraint was not met due to some assumptions during chemical reactions. The thermal analysis using DCS of the reaction of the new synthesized salt is endothermic where an amount of heat released and used for air heating.

VII. References

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VIII. Appendix

The designed system can be seen as follows:

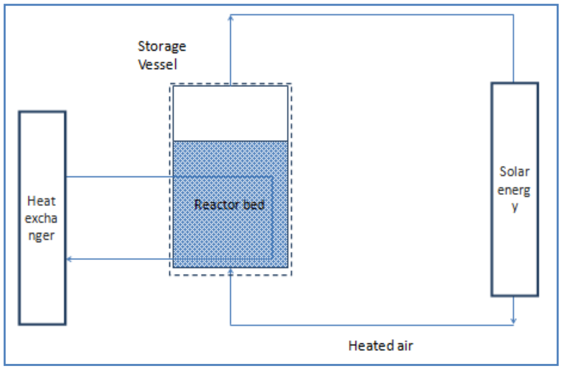

Figure 10: overall diagram of the storage vessel.

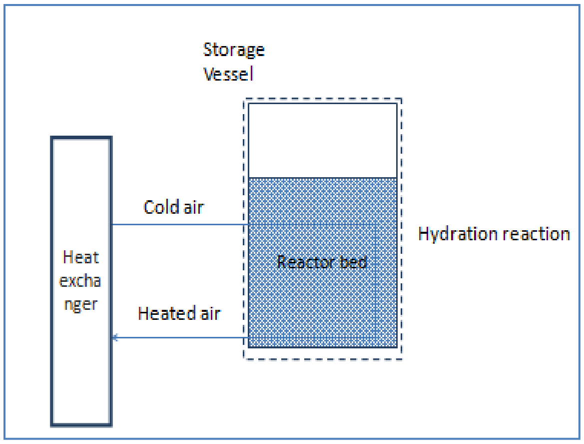

Figure 11: Hydration reaction.

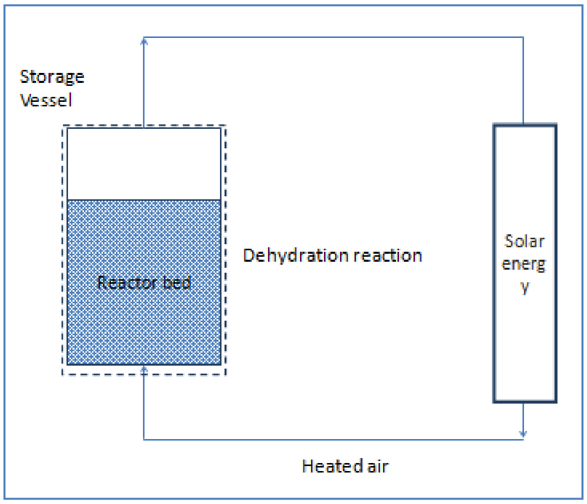

Figure 12: Dehydration reaction.

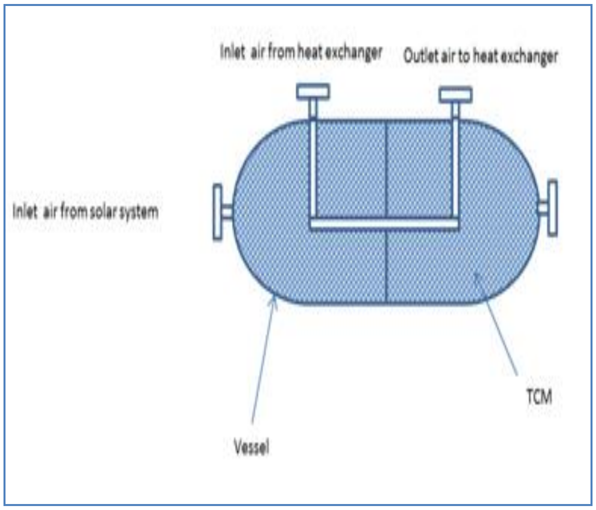

Figure 13: Vessel layout.

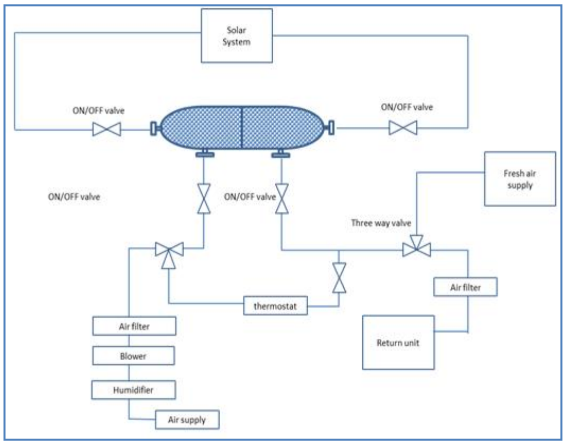

Figure 14: first suggestion of heat distribution system.

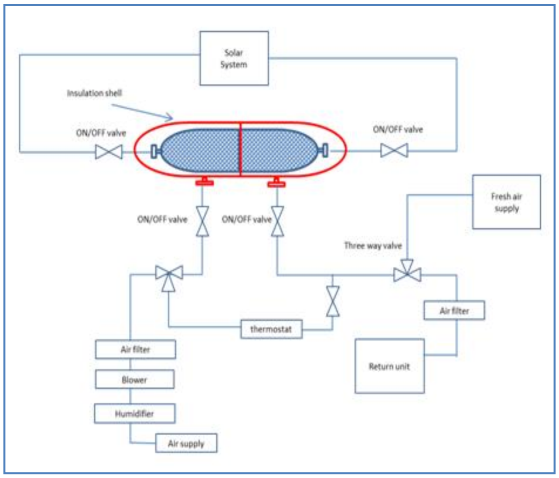

Figure 15: second suggestion.

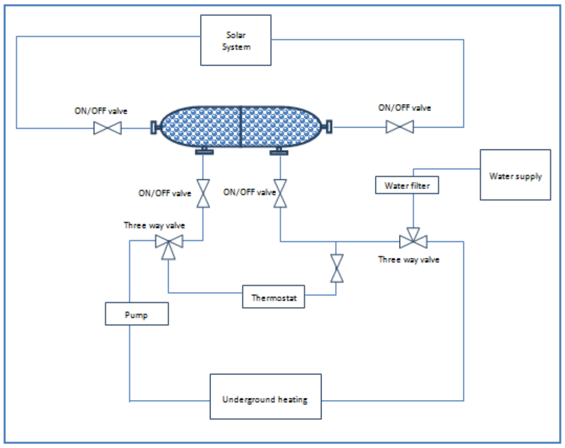

Figure 16: third suggestion.