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Macroencapsulation of Phase Change Materials for Thermal Energy Storage

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by

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ABSTRACT

The use of a latent heat storage system using phase change materials (PCMs) is an effective way of storing thermal energy. Latent heat storage enables high-energy storage density which reduces the footprint of the system and the cost. However, PCMs have very low thermal conductivities making them unsuitable for large-scale use without enhancing the effective thermal conductivity. In order to address, the low thermal conductivity of the PCMs, macroencapsulation of PCMs has been adopted as an effective technique. The macroencapsulation not only provides a self-supporting structure of PCM and separates the PCM from thermal fluids but also enhances the heat transfer rate.

The current work involves study of various concepts of encapsulation of low cost inorganic PCMs. Sodium nitrate ($NaNO_3$), a low cost PCM, was selected for thermal storage in a temperature range of 300 – 500˚C. Various techniques like electroless coatings, coatings using silicates, coatings with metal oxide (SiO_2) and sand encapsulation are discussed. A novel technique of metal oxide coating was developed where firstly a high temperature polymer, such as, polymer (stable > 500˚C) was coated over PCM pellets, and cured, so that the pellet becomes insoluble in water as well as several organic solvents and later the metal oxide is coated over the pellet using self-assembly, hydrolysis, and simultaneous chemical oxidation at various temperatures. The coated PCM pellets were characterized.

CHAPTER 1: INTRODUCTION

1.1 General Background

The economic and technological development worldwide leads to an increasing energy demand. However, conventional fossil energy sources are limited, and their use results in emission of harmful gases, which are responsible for climate changes and environmental pollution. An abundant renewable energy source is essential for reducing dependency on the fossil fuels and contributing to a cleaner environment. Solar energy is an essentially inexhaustible source potentially capable of meeting a significant portion of the nation's future energy needs with a minimum of adverse environmental consequences [1]. Solar energy is one of the most promising of the unconventional energy sources.

Solar radiation is intermittent by its nature. It varies as a factor of time, weather condition and latitude. If no energy storage is used in solar energy systems, a major part of the energy demand will be met by the back-up or conventional energy and therefore the annual solar load fraction will be very low. Solar energy storage will act as a panacea to all problems of energy conservation and levels the energy demand patterns. Among various energy storage techniques, thermal energy storage (TES) can be considered as one of the effective ones [2].

For solar energy to become an important energy source, efficient, economical and reliable, solar thermal energy storage devices and methods will have to be developed. The storage of energy is a present day challenge to the technologists. Energy storage not

only reduces the mismatch between supply and demand but plays an important role in conserving the energy. It leads to saving of premium fuels which are limited and also contributes to a green environment.

TES can be classified as latent, sensible and thermochemical energy storage. Latent heat refers to the heat released or absorbed when a material changes its phase at constant temperature. Latent heat energy storage contains PCMs and possess advantages like high energy storage density and small temperature difference between charging and discharging [3]. This helps in reducing the footprint of the system and the cost.

The key element in a latent heat thermal energy storage system is the storage material or the PCM. There are extensive studies on various kinds of PCMs like salts, paraffin waxes, inorganic acids, eutectics of organic and inorganic compounds [4-9]. Amongst them inorganic compounds come with advantages like high latent heat per unit volume, high thermal conductivity, non-flammability and low cost in comparison to organic compounds [9]. In the current work sodium nitrate ($NaNO_3$) is used as the PCM.

The application of inorganic materials for heat storage is accompanied with limitations like low thermal conductivity (0.1- 0.6 W/m K) that leads to low heat transfer and problem of oxidation on exposure to heat transport medium (air or heat transfer fluids like oils). In order to overcome these problems certain heat transfer enhancement techniques concepts such as use of extended surfaces, employing multiple PCMs, dispersion of high conductivity materials, etc. in heat exchangers have been identified [10-14]. The work presented in this thesis discusses the development of effective

techniques to encapsulate the PCM within a supporting structure and incorporating in a heat exchanger.

Macroencapsulation refers to a technique where a significant quantity of PCM is encapsulated as a discrete unit. It aids in holding the liquid PCM during phase change by providing a self-supporting structure, improves heat transfer rate and preserves the material composition [15]. Until now, macrocapsules were manufactured through containment method which comprises the inclusion of PCM in some form of package such as tubes, pouches, spheres, panels, tin-plated metal cans and mild steel cans or other receptacle [15, 16]. These containers can be incorporated in the heat exchangers.

1.2 Objective of Current Work

Several forms of macroencapsulated PCMs were prepared and marketed in the past two decades. Some of the commercially manufactured phase change heat storage products include TEAP Polyolefine spherical capsules, TEAP Polypropylene flat panel, EPS Ltd stainless ball capsule, EPS Ltd module beam, encapsulation in bags from Climator, Sweden and Dorken, Germany [17]. The most cost-effective containers available in the current market are "plastic bottles (high density and low density polyethylene bottles, polypropylene bottles), tin-plated metal cans and mild steel cans" [15].

The objective of the current work is to encapsulate the low cost PCMs such as inorganic salts ($NaNO_3$, KNO_3, or in mixture etc.) for a storage system in a temperature range of 300 – 500˚C. $NaNO_3$ is used owing to the advantages of its low cost and significantly high latent heat compared to other PCMs that melt at similar temperatures. Different

techniques like electroless coatings, metal oxide and polymer coatings, using water glass and sand encapsulation were studied. The scope of current work encompasses:

1. Preparation of the PCM pellets
2. Encapsulation of PCMs using various techniques
3. Optimizing the encapsulation techniques for obtaining the best results so that the capsules withstand greater number of thermal cycles
4. Characteristic evaluation of the encapsulated pellets (metal oxide and polymer coatings).

The entire work is presented as four chapters in this thesis and the gist of them is provided below.

Chapter 1 provides an introduction which portrays the importance of solar energy and gives general overview of the research work.

Chapter 2 gives a literature review on the thermal energy storage, performance enhancement techniques, PCMs and encapsulation of PCMs in latent heat thermal energy storage systems (LHTES).

Chapter 3 includes preparation of PCM pellets and various methods adopted for encapsulating the PCMs at macro-level and their outcomes. It describes the novel method of encapsulation by depositing a metal oxide over PCM and the characterization study performed on the coatings.

Chapter 4 gives summary and conclusion of the current work. Recommendations for future work.

CHAPTER 2: LITERATURE SURVEY

2.1 Thermal Energy Storage

Solar energy is one of the most abundantly available sources of renewable energy. Since its intermittent in nature, efficient utilization of this substantial resource requires proper storage technology. The solar energy storage in the form of thermal energy has gained lot of attention because of the potential outcome it can deliver to address the growing energy needs.

The design criteria in building a TES system can be classified as [18]:

1. Cost Criteria: It includes selection of PCM and the heat exchanger mainly affects the cost of the system. Space and installation costs account to the total cost.
2. Technical Criteria: It includes selection of PCM with high storage capacity and stability, ensuring good heat transfer between heat transfer fluid and the PCM, extent of thermal losses, load capacity, operational temperature, reduction in specific enthalpy of load upon long operation and integration into the power plant.

2.2 Forms of Thermal Energy Storage

Thermal energy can be stored as a change in internal energy of a material as sensible heat, latent heat and thermochemical or combination of these [19]. Major TES techniques are shown in Figure 1 [19]. A detailed discussion of the storage techniques is presented below.

2.2.1 Storage Using Sensible Heat

Sensible heat storage is achieved by raising the temperature of a liquid medium (water, oil-based liquids, molten salts etc.) or a solid medium (like rocks, metals, and others) [1, 20-22]. In such a system the amount of heat stored depends on mass, specific heat and the temperature change within the storage material and is given by:

$$Q_{sensible} = \int_{T_1}^{T_2} mC_p \, dT \qquad \text{Eq. (1)}$$

Currently installed TES systems in solar thermal plants store sensible heat energy. They use two-tanks with either oil or molten salt. Both oil and molten salt systems were found to be technically feasible.

Important considerations like costs, total system investment, very large tank size requirements, and inflexibility, compared to a back-up system, made oil usage infeasible in solar electric generating system plants.

Use of molten salts has been demonstrated in the 10 MW Solar Two project and is now being considered by companies in Spain, Italy and USA. The salts, however, generally have a high melting point and parasitic heating is required to keep them liquid at night, during low insolation periods, or during plant shutdowns. The higher outlet temperature comes with heat losses and requires more expensive piping and materials.

Alternate concepts that were considered by researchers include sensible heat storage in solid media like concrete. The advantage of concrete systems is the low cost of the thermal energy storage medium. The disadvantages are large volumes, increased costs of heat exchangers and engineering.

2.2.2 Storage Using Latent Heat (LHTS)

Storage systems based on phase change materials can be smaller, more efficient and a lower cost alternative to sensible thermal storage systems. There have been many studies on solar thermal energy storage using PCMs [7, 23-30]. Of the different forms of phase change processes, the solid –liquid transition is efficient in terms of low volumetric expansion compared to the liquid – gas transition and high latent heat compared to the solid – solid transition. From an energy efficiency point of view, PCM storage systems have the advantage that they operate with small temperature differences between charging and discharging as shown in Figure 2 and possess high energy density compared to sensible heat storage. The amount of heat stored in a latent heat storage system is given by:

$$Q_{latent} = \int_{T_1}^{T_m} mC_p\, dT + mL + \int_{T_m}^{T_2} mC_p\, dT \qquad \text{Eq. (2)}$$

Several types of PCMs are available based on the type of application. For example, for melting ranges between 0˚C to 200˚C, PCMs such as paraffins, fatty acids, polymers, salt hydrates and sugar alcohols may be used. At higher melting temperatures, salts, eutectics, high performance polymers, metal alloys and carbonates are available.

2.2.3 Storage Using Chemical Reactions

Storage by means of chemical reactions has also been considered by many researchers for a wide range of temperatures [31, 32] using reversible endothermic/exothermic reactions. Heat stored in a chemical reaction depends on mass of storage material, endothermic heat of reaction and extent of reaction and is given by:

$$Q_{chemical} = m a_r \Delta h_r \quad \text{Eq. (3)}$$

Drawbacks may include complexity, uncertainties in the thermodynamic properties of the reaction components and of the reaction kinetics under a wide range of operating conditions, high cost, toxicity, and flammability.

Implementation of various storage technologies in CSP plants suffers from the aforementioned tailbacks. There is a dearth of cost effective solutions to deal with the problems posed by various energy storage technologies. Hence, there is a need for developing an efficient TES system for achieving a cost-effective solution for successful implementation in solar power plants on a large scale. Based on the review of the available options, PCM storage can provide a cost effective solution, provided the heat transfer enhancement can be achieved at low cost.

2.3 Latent Heat Storage Materials

The PCMs are the energy storage materials which store heat by changing the phase at almost constant temperatures. This enables them to have considerably higher thermal energy storage densities compared to sensible heat storage materials. The PCMs operate by absorbing heat (charging) and increasing in temperature until phase change temperature, heat absorption at phase change from solid to liquid (in current application) occurs at almost constant temperature and the same process repeats with release of heat during the cooling (discharging) process and liquid completely converts into solid [33]. The main criteria that govern the selection of phase change heat storage materials are [8]:

1. Melting point should be in the operating temperature range of TES system.

2. High latent heat of fusion per unit mass, which ensures high energy density
3. High specific heat to provide additional significant sensible heat storage effects.
4. High thermal conductivity in both solid and liquid phases, so that the temperature gradients for charging and discharging the storage material are small.
5. Small volume changes during phase transition, as it reduces size of encapsulate and ultimately the heat exchanger.
6. Exhibit little or no subcooling during freezing.
7. Possess high density
8. High chemical stability especially at higher temperatures, no chemical decomposition upon extended cycling and reaction resistance to encapsulating materials
9. Should be non-poisonous, non-flammable and non-explosive elements/compounds in the entire operating temperature range
10. Low cost and abundance

The PCMs can be organic, inorganic, fatty acids etc., and the selection of PCM varies according to the engineering application and its requirements [34]. In the TES systems, PCMs with higher energy storage density are preferred as they require less volume and hence reduce the system costs [15]. There are extensive studies on PCMs by Lane [4], Hasnain [35], Mohammed Farid [9], Zalba [7] and Sharma [19].

However, most PCMs have low thermal conductivity, and that leads to slow charging and discharging rates [36]. The effective thermal conductivity can be improved by [4]:

1. Adding materials with high thermal conductivity to the pure PCM.
2. Microencapsulation of PCM in the metallic or non-metallic (graphite) matrix, [9]

3. Forming small macrocapsules of PCM and enhancing convective heat transfer by submerging the PCM capsules in a liquid.

Other methods [10-14] employed to enhance the heat transfer include, using extended surfaces, employing multiple PCM's, thermal conductivity enhancement using metallic structures, PCM impregnated foams, dispersion of highly conductive particles and encapsulation of PCM. A brief review of such techniques is presented in the following section.

2.4 Performance Enhancement of Latent Heat Thermal Storage System

Though latent heat thermal energy storage technique has various advantages like large energy storage for a given volume, uniform energy storage/supply and compactness, the large-scale utilization is still not in progress. The main reason behind the failure is low thermal conductivity of the PCM which results in significant temperature drop during charging and discharging processes. All conventional PCMs have very low thermal conductivity ranging from 0.1 to 0.6W/mK. Hence it becomes vital to ensure the enhancement of thermal performance of the LHTS system. A great deal of theoretical and experimental work was done in this direction. The various enhancement techniques as summarized by Jagadheeswaran [10] are given below and briefly discussed further.

1. Using extended surfaces
2. Employing multiple PCM's method
3. Thermal conductivity enhancement
4. Encapsulation of PCM

2.4.1 Extended Surfaces

Extended surfaces, generally fins are embedded in the PCM to provide an additional heat transfer surface. This method comes with advantages of simplicity, ease in fabrication and low cost of construction. Fins can be in any shape like circular, longitudinal etc., and shown in Figure 3 [8]. As the efficiency of fins increases with the decrease in heat transfer coefficient, fins are usually placed in the PCM rather than in the HTF. The performance enhancement that can be achieved through fins depends on configuration and orientation of the thermal system and fins [10].

Many studies have illustrated it both numerically and analytically by considering PCM enclosed in rectangular, cylindrical and spherical enclosures. The presence of fins in the PCM enhances the melting rate by improving the natural convection in case of large containers. It was also suggested that length of the fins and the number of fins are important design factors. The optimum number of fins depends on the wall temperature, beyond certain number the increase in heat transfer rate was marginal due to the hampering effect on the buoyancy driven flows. The fins help in increasing the solidification rate by enhancing the conduction heat transfer and melting rate by enhancing convection heat transfer. The number of fins required to be employed should be optimized with respect to energy stored and performance enhancement required [10].

2.4.2 Employing Multiple PCMs

Employing multiple PCMs in LHTS system is another performance enhancement technique where the system is packed with more than one PCM of different melting temperatures. The idea behind this concept is to maintain nearly constant temperature

difference between HTF and the molten PCM during charging and discharging of the system. This wouldn't be possible in case of a single PCM as there would be a decrease in temperature difference in the flow direction, which deteriorates the system performance by bringing down the heat transfer rate. Since the heat transfer rate mainly depends on the difference between HTF and the melting point of PCM, multiple PCMs with decreasing order in their melting points are packed together to maintain a nearly constant temperature difference. During discharging, if the HTF flow direction is reversed in a way that the PCMs remain in the increasing order of their melting points then nearly constant heat flux from the PCM to HTF is possible. Such an illustration of use of multiple PCMs in shell and tube heat exchanger is given in Figure 4 [10].

This method can be more efficient if the homogeneous phase change process is possible, i.e., phase change takes place everywhere in the PCM. But, in reality such combination is difficult to achieve. Besides appropriate difference between melting points and relative proportions of PCMs, issues also arise with compatibility among the PCMs. As the studies performed till now are done by choosing arbitrary combination of PCMs, more research has to be done before applying this concept for obtaining best performance enhancement results. Studies also suggest that employing multiple PCMs in conjunction with extended surfaces could provide much better results. However, a more profound investigation is yet to be performed in this direction [10].

2.4.3 Thermal Conductivity Enhancement

One of the potential techniques to enhance the thermal conductivity of conventional PCMs is to employ high conductivity materials and can be summarized in the following ways [10]:

1. Impregnation of high conductivity porous material with the PCM
2. Dispersion of high conductivity particles in the PCM
3. Placing of metal structures in the PCM
4. Use of high conductivity, low density materials

The detailed description of these techniques is given below.

2.4.3.1 Impregnation of Porous Material

Porous materials include either metal matrix made of aluminium, copper, diamond coated copper, graphite, etc. Numerical studies revealed that impregnation of such materials result in effective increase in thermal conductivity of the system. The performance enhancement by the impregnation was dependent on both porosity and thermal conductivity of the matrix. The porosity plays an important role since it affects the natural convection within the PCM [10].

Most of the studies done in this direction were using different forms of graphite owing to its favorable features like high thermal conductivity, high electrical conductivity and high absorbability. Graphite, as shown in Figure 5, was available in many forms like cheapest natural form, expanded graphite, exfoliated graphite nano pellets, ground expanded graphite powder. The PCMs considered were paraffin and inorganic salts. The composites with paraffin were prepared by utilizing the capillary and surface tension

forces. The composites with inorganic PCMs were prepared either by compounding, i.e., adding graphite to the molten salts at high temperature and mixing them or by cold compression, where solid PCM and graphite are mixed together and them compressed into a mold under a press. The later method was more economical and was free from corrosion effects and safety issues but it possessed anisotropic characteristics arising from rearrangement of graphite layer orthogonal to compression axis [10].

The studies also included the effect of amount and size of graphite added in the PCM. It was found that the composite PCM possessed almost a congruent melting point with a slight decrease as compared to the original phase change temperature [10].

2.4.3.2 Dispersion of High Conductivity Particles in the PCM

Inspite of many advantages possessed by impregnation of graphite, a major drawback was discovered which relates to small mean pore size that leads to decrease in latent heat by hindering of molecular motion. This drawback accompanied with other disadvantages like lengthy preparation processes gave room for an alternative technique of dispersion of high conductivity particles to enhance the thermal conductivity of PCM [10].

Micro aluminium, copper and silver particles were also considered by researchers and studied for obtaining composite PCMs. Amongst the considered particles silver was comparatively inert to the salts. The dispersed particles reduced the volume of the PCM and stimulated the solidification of the nanofluid. Hence the mass fraction of particles to be added to enhance the performance of the system was an optimization between heat storage and thermal conductivity [10].

2.4.3.3 Placing of Metal Structures

Another technique reported in literature for enhancing the thermal conductivity was placing of metal structures into the PCMs. Some of the metal structures included were thin walled hollow cylindrical steel structures called lesser rings (shown in Figure 6 [29]), stainless steel balls combined with stainless steel screens and steel balls inserted into the PCM stored in small spherical module. It was observed experimentally that enhancement due to metal structures depends purely on their number and the dimensions and to get the same reduction in solidification time, more volume of metal structures were required as compared to that of fins. Also the thermal conductivity improvement due to metal structures depends upon the diameter of the cylindrical LHTS module, whereas in case of fins, it remains almost constant for a fixed number of fins making the metal structures more effective than fins in larger storage systems [10].

2.4.3.4 Use of High Conductivity and Low Density Materials

Owing to the relatively high density of the metal particles/metal structures, the weight of the system increased and moreover compatibility issues with PCM prevailed as well. Hence, the investigations were also probed into low density materials like carbon fibers which have thermal conductivities almost equal to metals. Besides being compatible with most of the PCMs, carbon fibers have comparatively more corrosion resistance. It was also observed that enhancement performance depends highly on the uniform distribution of the fibers in the PCM. A system with uniformly distribution of the carbon fibers like bush type and fiber cloths, shown in Figure 7 [37], [10] in the heat flow direction was found to be more effective compared to the one with random distributed fibers [10].

It was also reported that the heat transfer rate increased with the increase in brush diameter, but beyond certain diameter the known as critical diameter the heat transfer rate remained almost constant. It was due to crossing of fibers beyond that diameter which led to formation of low density areas, which in turn hindered heat transfer rate. Studies also revealed carbon brushes to be economically viable taking reduction of size of LHTS unit into consideration [10].

2.4.4 Encapsulation of PCMs

The process of encapsulation increases the heat transfer area and thereby heat transfer coefficient of the system. It is also helpful in forming a barrier and protecting the PCM from the outside environment and controlling the volume changes of the PCM [38]. Encapsulated PCMs are composed of PCMs as core and polymer or inorganic material as shell to maintain the shape and prevent PCM from leakage during phase change process. Depending on the size, the encapsulated PCMs are classified as NanoPCMs, MicroPCMs and MacroPCMs. The size of MicroPCMs typically varies from 1 μm to 1 mm, while the capsules smaller than 1 μm are classified as NanoPCMs and capsules larger than 1 mm as MacroPCMs [39]. Owing to the advantages like reduction of the reactivity of the PCMs with outside environment, increase the heat transfer area and provision for the core material to withstand changes in volume of the PCM, as the phase change occurs they have attracted considerable interest in the fields of energy storage like solar energy storage utilization, thermo-regulated fibers and foams, energy-saving building materials, etc. [39].

2.5 Encapsulation of PCMs in Latent Heat Thermal Energy Storage

The heat transfer rate between PCM and source/sink can be increased by using encapsulated PCMs. It has developed interest in several researchers especially because it can be cost effective comparative to other techniques. There are mainly two different means of encapsulation.

The first is microencapsulation, whereby small, spherical or rod-shaped particles are enclosed in a thin, high molecular weight polymeric film. Hence, the microencapsulated PCMs contain micro size PCM in liquid/solid (core) form enveloped within a solid structure (shell/wall). The shell can be made of wide range of materials including natural and synthetic polymers and must be compatible with PCM. The microscope profiles of microencapsulated PCMs obtained from spray drying and coacervation methods can be seen from Figure 8 [10], [40].

The second containment method is macroencapsulation, which comprises of PCM in some form of self-assembled structure or a package such as tubes, pouches, spheres, panels or other receptacle. These containers usually are larger than 1 cm in diameter and can be incorporated in building products as well. The main advantage of the macroencapsulation is its applicability to both liquid and air as heat transfer fluids and easier to ship and handle. Until now, the most cost-effective containers are plastic bottles (high density and low density polyethylene bottles, polypropylene bottles), tin-plated metal cans and mild steel cans [15]. Some of the commercially manufactured macrocapsules are shown in Figure 9 [5]. In the case of microencapsulation, unless the matrix encapsulating the PCM has high thermal conductivity, the microencapsulation

system suffers from low heat transfer rate. The rigidity of the matrix prevents convective currents and forces all heat transfer to occur by conduction. This can reduce seriously the heat transfer rates, especially in the charging mode. Besides holding the liquid PCM and preventing changes of its composition through contact with the environment, macroencapsulation also improves the ease of handling PCM and its compatibility with the surroundings [17].

Coating materials used in the encapsulation of PCMs should meet the following characteristics:

1. Have high strength, flexibility and thermal stability;
2. Stable to UV exposure, barrier to moisture, air, etc;
3. Stable to environmental conditions;
4. Capable of being used safely;
5. High thermal conductivity;
6. Not corrosive to container materials;
7. No migration of PCMs into coating materials;
8. No reaction between PCMs and coating materials.

Successful utilization of encapsulated PCM heat storage media depends on developing means of containment. Currently, extensive research is being done in developing macroencapsulation technologies using a cost effective materials and methods of production. Some examples of published work on encapsulated PCM systems, along with the materials used, preparation method and their intended applications are shown in Table 1 [41].

A great deal of work also proceeded in conserving energy with thermal storage by using latent heat from PCMs. Amar M. Khudhair [16] summarized the investigation and analysis of thermal energy storage systems incorporating PCMs for use in building applications and researches on thermal storage in which the PCM is encapsulated in concrete, gypsum wallboard, ceiling and floor that have been ongoing were discussed. Cabeza [17] compiled information about the requirements of the use of TES technology, classification of materials, materials available and problems and possible solutions on the application of such materials in buildings.

Zhou [7] also summarized previous works on latent thermal energy storage in building applications, covering PCMs, the impregnation methods, current building applications and their thermal performance analyses, as well as numerical simulation of buildings with PCMs.

Presently the cost of the microencapsulation system is high compared to other storage methods, and is used only in thermal control applications. Macrocapsule is the dominant and potential containment system for PCM [15] and can be employed for wide variety of energy storage needs.

2.6 Heat Storage Tanks

In order to use heat storage products, an appropriate heat exchange device should be designed for each particular application. However, in practice, companies produce a set of typical standardized units. Table 2 [5] presents information on dimensions and volumes of storage tanks produced by Cristopia and EPS.

2.7 Scope of the Present Work

In a latent heat storage system that uses a high latent heat phase change material (PCM), macroencapsulation of the PCM helps to overcome the barrier of low thermal conductivity by increasing heat transfer rate, provide the separation of the PCM from other fluids where needed and offer a self-supporting structure for the PCM.

In a LHTES system (as shown in Figure 10) the heat is transferred to or from a heat transfer fluid as the heat transfer fluid flows through the space between the capsules. During the charging mode, the hot fluid carrying energy from the solar field is circulated through the tank. The PCM inside the capsules absorbs latent heat and melts. During the discharging mode, cooler heat transfer fluid is circulated through the tank to absorb heat from the PCM resulting in freezing of the encapsulated PCM. The heated fluid is then used to heat the power block working fluid through a heat exchanger.

The major challenges to the success of the above described concept are:

1. Forming porous macro-spheres of the PCM material at optimum size and the optimum pore volume in the macro-spheres that will account for the volume change from solid to liquid phase.
2. Encapsulating the macro-spheres of PCM in a higher melting temperature material, in such a way that it gives required strength and stability to hold the PCM over extended cycling.

The performance and efficiency of the encapsulation coatings is verified through heating and cooling cycles. The cyclic process that is used for testing the encapsulated pellets in the furnace is shown in Figure 11.

The focus of this work is to address the above mentioned challenges and fabricate encapsulated pellets which can withstand a greater number of thermal cycles.

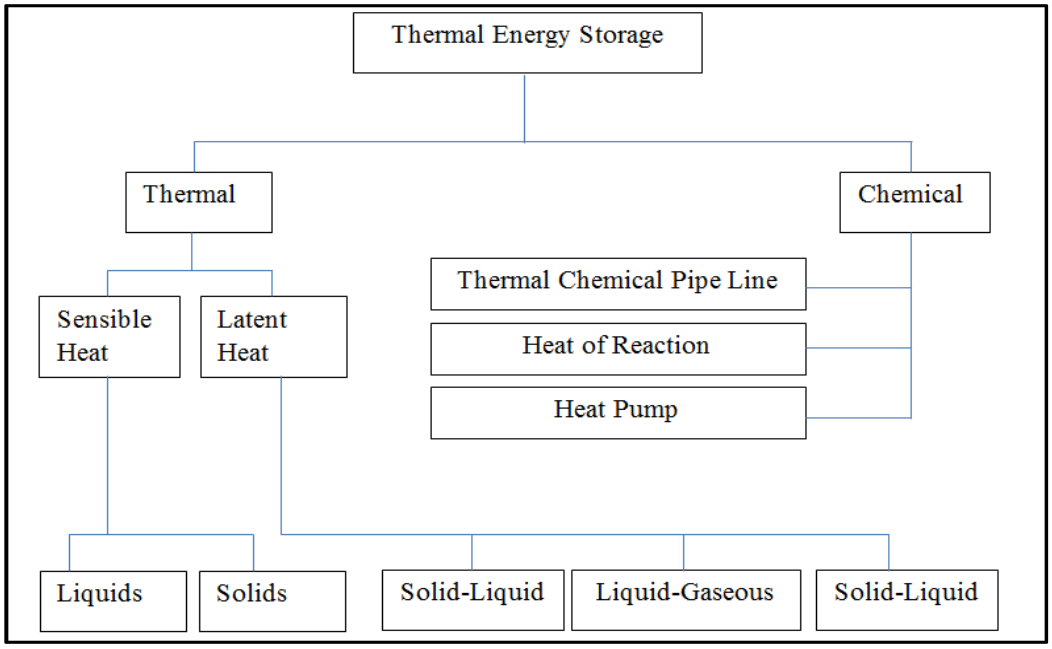

Figure 1: Different types of thermal storage of solar energy [19] [1]

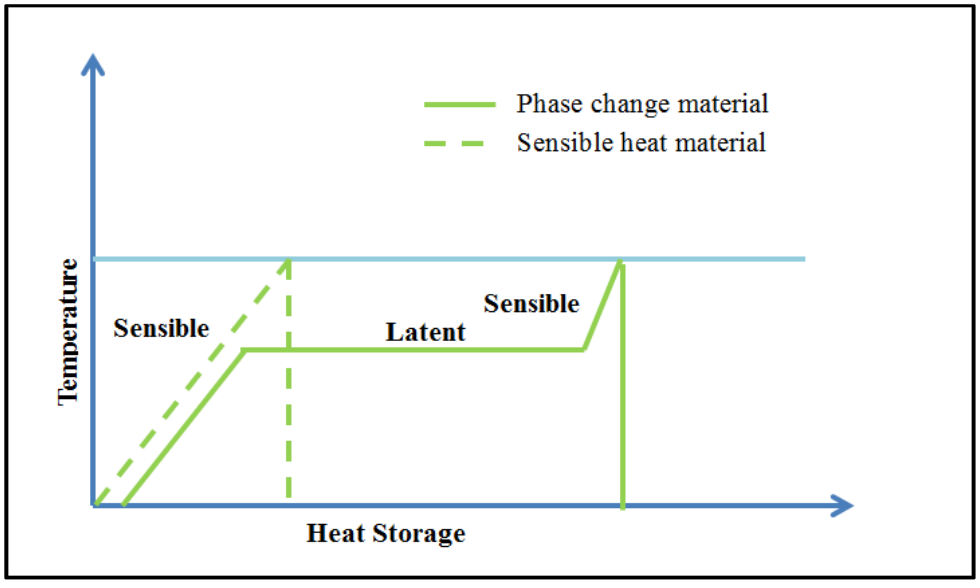

Figure 2: Heat storage comparison between a sensible heat material and a PCM

[1] Figure 1 is reprinted from Renewable and Sustainable Energy Reviews, 13(2), Sharma, A., et al., Review on thermal energy storage with phase change materials and applications, p. 318-345., 2009, with permission from Elsevier

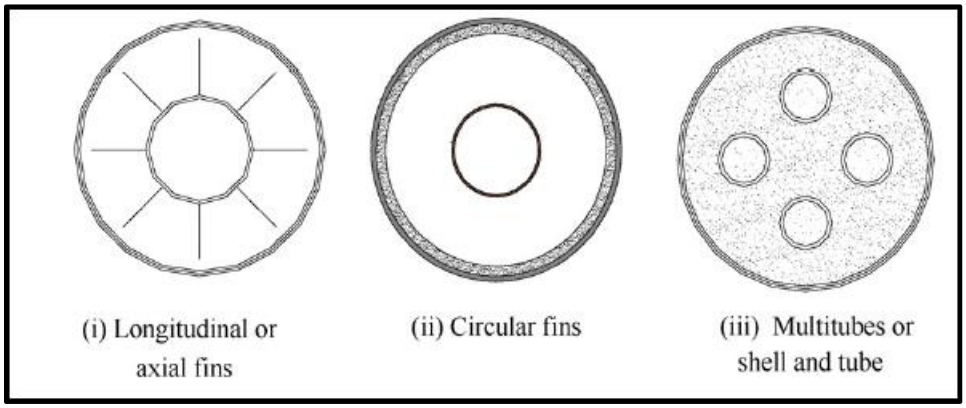

Figure 3: Extended surfaces [8] [2]

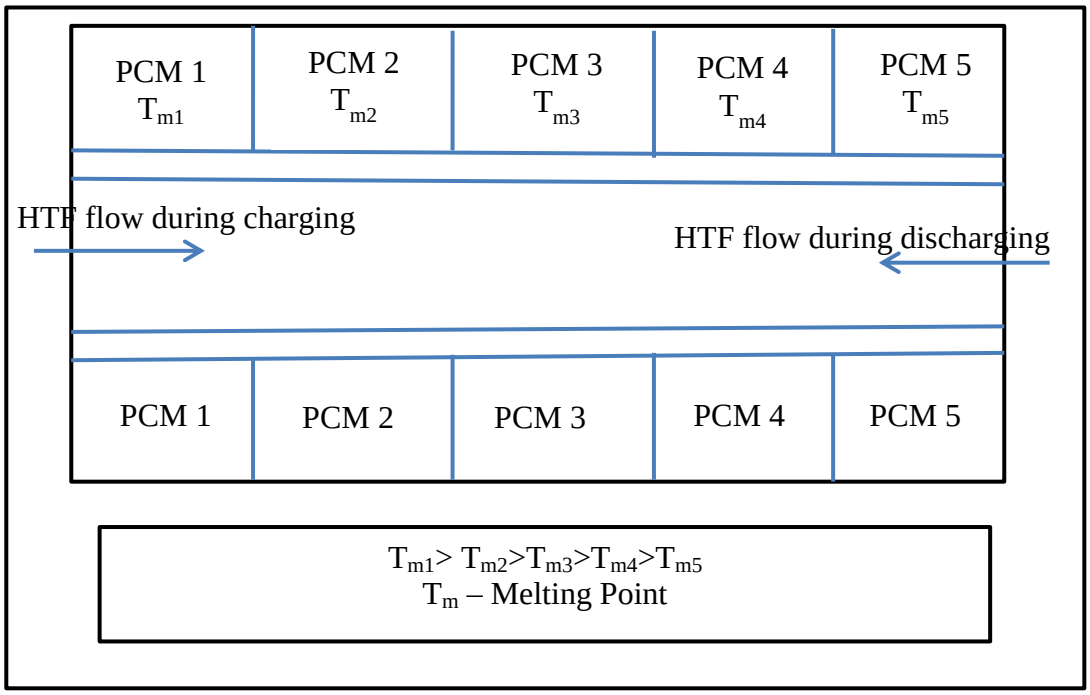

Figure 4: Multiple PCMs in shell and tube LHTS unit [10][3]

[2] Figure 3 is reprinted from Renewable and Sustainable Energy Reviews, 14(2), Agyenim, F., et al., A review of materials, heat transfer and phase change problem formulation for latent heat thermal energy storage systems (LHTESS). p. 615-628, 2010 with permission from Elsevier

[3] Figure 4 is reprinted from Renewable and Sustainable Energy Reviews, 13(9), Jegadheeswaran, S. and S.D. Pohekar, Performance enhancement in latent heat thermal storage system: A review, p. 2225-2244., 2009 with permission from Elsevier

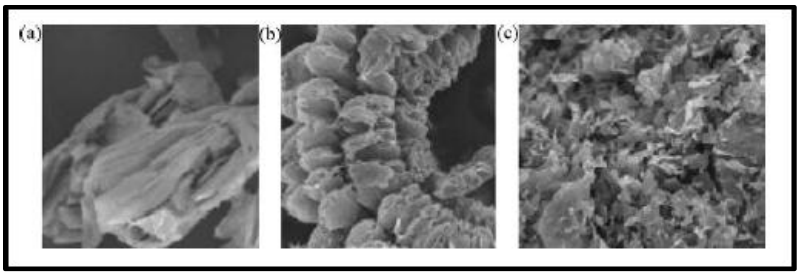

Figure 5: Types of graphite [10][4]

Figure 6: A photographic view of lesser rings [29][5]

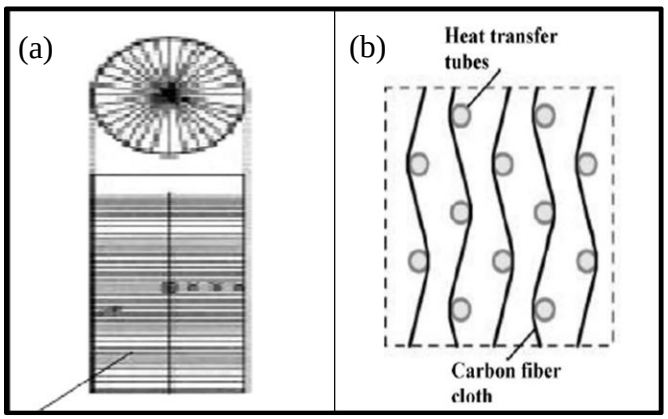

Figure 7: Carbon fibers: (a) carbon fiber brushes; (b) carbon fiber cloth [10], [37] [6]

[4] Figure 5 is reprinted from Renewable and Sustainable Energy Reviews, 13(9), Jegadheeswaran, S. and S.D. Pohekar, Performance enhancement in latent heat thermal storage system: A review, p. 2225-2244., 2009, with permission from Elsevier

[5] Figure 6 is reprinted from Solar Energy, 65(3), Velraj, R., Seeniraj, R. V., Hafner, B. Faber, C., Schwarzer, K., Heat Transfer Enhancement in a Latent Heat Storage System, p. 171-180., 1999, with permission from Elsevier

[6] Figure 7 is reprinted from Energy Conversion and Management, 41(14), Fukai, J., et al., Thermal conductivity enhancement of energy storage media using carbon fibers, p. 1543-1556., 2000, & Renewable and Sustainable Energy Reviews, 13(9), Jegadheeswaran, S. and S.D. Pohekar, Performance enhancement in latent heat thermal storage system: A review, p. 2225-2244., 2009, with permission from Elsevier

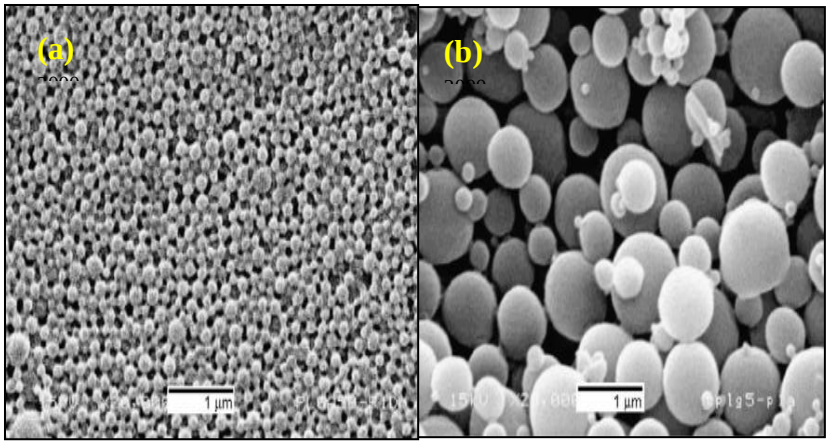

Figure 8: Microscopic profiles of microencapsulated PCMs (a) from spray drying method; (b) from coacervation method [10], [40][7]

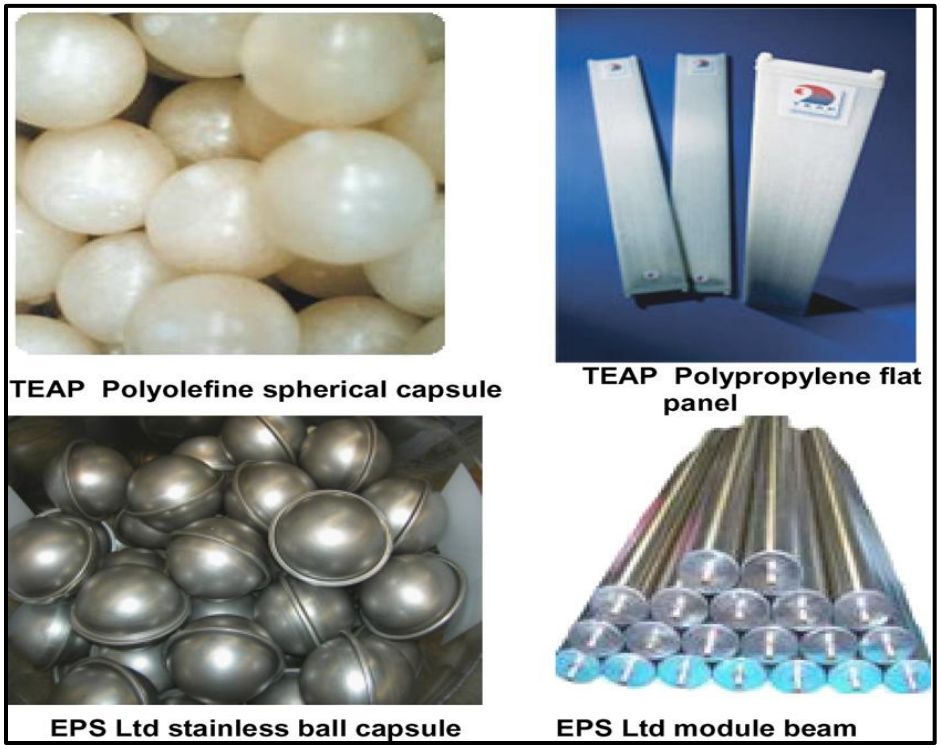

Figure 9: Commercially manufactured macrocapsules [5][8]

[7] Figure 8 is reprinted from Applied Energy, 74(1–2), Hawlader, M.N.A., M.S. Uddin, and M.M. Khin, Microencapsulated PCM thermal-energy storage system. p. 195-202., 2003, & Renewable and Sustainable Energy Reviews, 13(9), Jegadheeswaran, S. and S.D. Pohekar, Performance enhancement in latent heat thermal storage system: A review, p. 2225-2244., 2009, with permission from Elsevier

[8] Figure 9 is reprinted from Renewable and Sustainable Energy Reviews, 11(9), Kenisarin, M. and K. Mahkamov, Solar energy storage using phase change materials, p. 1913-1965., 2007, with permission from Elsevier

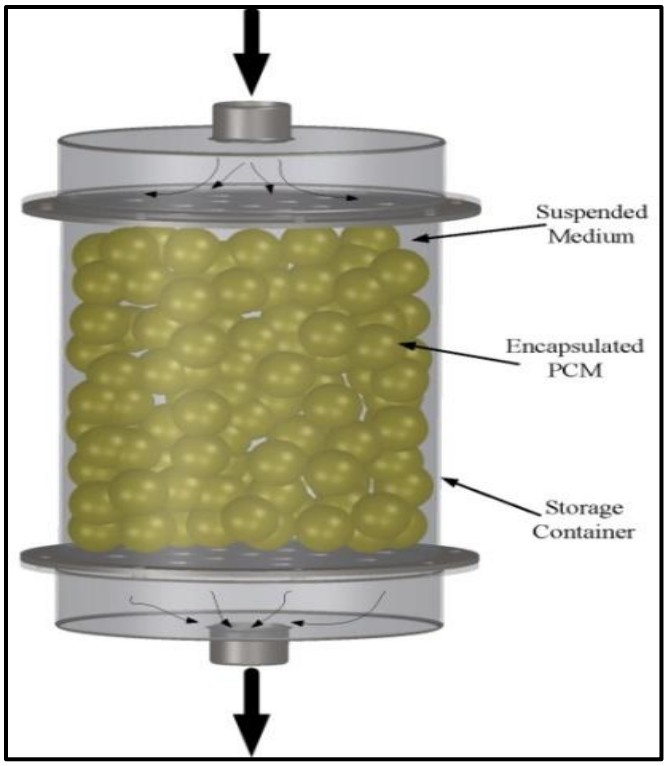

Figure 10: Direct contact TES system

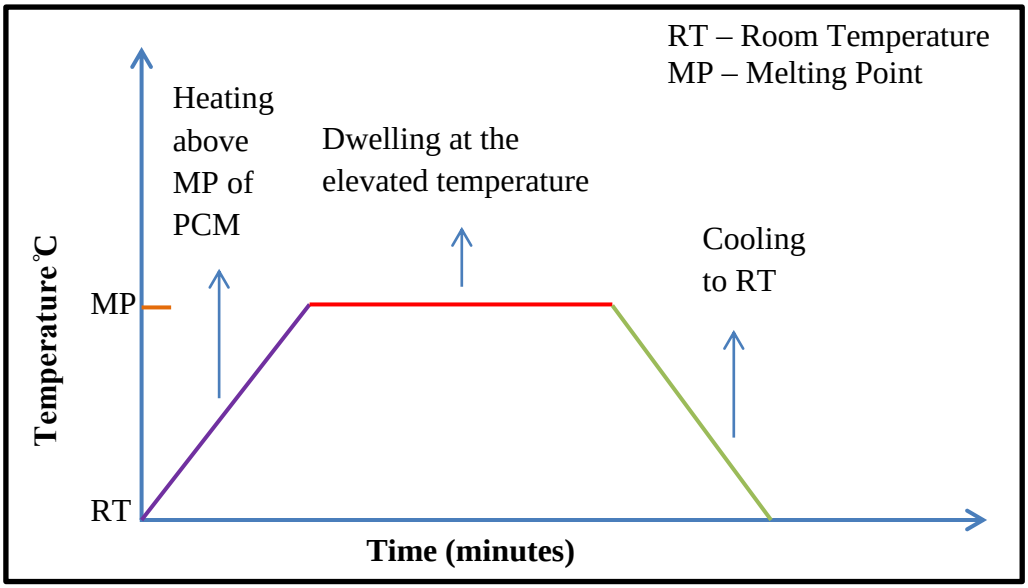

Figure 11: The thermal cycling

Table 1: List of a few published encapsulated PCM systems [41][9]

Investigators and year	Core materials	Coating materials used	Methods	Types of capsules	Applications
Lane, 1980 [42]	Eutectic $Mg(NO_3)_2.6H_2O$-$MgCl_2.6H_2O$	Polyester	Sealing with polyester film	Macrocapsules	Preheating domestic water in tank filled with encapsulated PCM
Morikama, 1985 [43]	Inorganic salt Hydrate	Polyester	Interfacial Polymerization	Matrix type Microcapsules	Thermal control in the buildings
Feldman1989 [44]	Organic PCM 30% wt, gypsum, cement, sawdust, sand and water	Polyester resin	Interfacial Polymerization	Floor, wall, ceiling tiles as composite materials	Storing offpeak electricity in home
Inaba et al., 1997 [45]	Paraffin wax	High density polyethylene	Interfacial Polymerization	Matrix type microcapsules	Integrating with building materials to reduce overheating in summer and to take effect storage discharge by ventilation
Brown et al., 1998 [46]	octadecane and paraffin	polymethyleneurea, cross-linked nylon, and gelatin	Interfacial Polymerization	Microcapsules	Gas-fluidized Bed
Salyer, 1999 [47]	Eutectic PCM	Polyester resin	Interfacial polymerization	Matrix type Microcapsules	Insulation in clothing

[9] A part of table 1 is taken from the thesis "Encapsulation of Phase Change Materials (PCMs) for Heat Storage" Department of Chemical and Environmental Engineering, National University of Singapore, 2003, with permission from Ms. Mya Mya Khin

Table 1 (Continued)

Xiao, 2000 [48]	Paraffin wax	Styrenebutadiene-styrene copolymer	Interfacial Polymerization	Matrix type Macrocapsules	Latent heat storage materials for thermal storage units
Hawlader, 2002 [49]	Paraffin wax	Gelatin and acacia	Complex Coacervation	Matrix type Microcapsules	Packed bed heat exchanger
Miroslaw Zukowski, 2005 (experimental study) [50]	Paraffin wax	polyethylene film bag	Enclosing PCM polyethylene film bags in 3 layers polyethylene film bag	Macro encapsules	TES applications for processes and buildings
Ahmet Sari, 2009 [51]	n-octacosane	polymethylmetracrylate (PMMA)	emulsion polymerization	Microcapsules	For potential energy storage
Guiyin Fang, 2009 [52]	n-Tetradecane	Urea and formaldehyde	in situ polymerization methods	Nanocapsules	thermal energy storage and heat transfer enhancement
Wei Li, 2011 [39]	n-Octadecane	Micro: gelatin-gum arabic shell, polyurethane shell and styrene-based copolymer shell Macro: calcium alginate	Micro: complex coacervation, interfacial polymerization and suspension polymerization Macro: piercing-solidifying incuber method	Micro and macrocapsules	Thermal energy storage and thermal regulated textiles and clothing

Table 1 (Continued)

Lin Pan, 2012 [38]	Palmitic acid	AlOOH	In situ emulsion interfacial poly-condensation method	Colloidal microcapsules	Thermal energy storage, enhancement

Table 2: Commercially manufactured phase change storage tanks [5][10]

Volume (m^3)	External diameter (mm)	Total Length (mm)	External surface area for insulation (m^2)	Connections inlet/outlet (mm)	Number of cardles	Empty weight (kg)	Heat transfer fluid volume (m^3)
Cristopia Energy Systems (Cristopia)							
2	950	2980	10	40	2	850	0.77
5	1250	4280	18	50	2	1250	1.94
10	1600	5240	29	80	2	1990	3.88
15	1900	5610	37	100	2	2900	5.82
20	1900	7400	47	125	3	3700	7.77
30	2200	8285	61	150	3	4700	11.64
50	2500	10,640	89	175	4	6900	19.40
70	3000	10,425	106	200	4	7300	27.16
100	3000	14,770	147	250	6	12,700	38.80
Environmental Process Systems Limited (EPS Ltd.)							
5	1250	3750	50				
10	1600	4500	80				
25	2000	8000	125				
50	2500	10,000	150				
75	3000	10,600	200				
100	3000	11,100	250				

[10] Table 2 is reprinted from Renewable and Sustainable Energy Reviews, 11(9), Kenisarin, M. and K. Mahkamov, Solar energy storage using phase change materials, p. 1913-1965., 2007, with permission from Elsevier

CHAPTER 3: DEVELOPMENT OF MACROENCAPSULATED PCMs

In order to improve the energy storage efficiency and reduce costs, proper design of the system is necessary. Selection of a PCM and associated encapsulation method is the first issue that must be addressed, for a practical application, because the efficiency of the thermal energy storage system primarily depends on the heat transfer and heat storage capability of the material. The cost of the TES system not only depends on the PCM material but also on the encapsulation and containment (tank etc.) and associated systems. Advantages and disadvantages of different geometries of PCM encapsulation with different materials and their compatibility were discussed [7, 9].

3.1 Selection of Material

Selection of PCM is a vital factor in determining the efficiency of the TES systems. PCM can be selected depending on the melting temperature, latent and sensible heat capacities, thermal stability, mechanical stability, withstand thermal cycling, heat transfer characteristics, and cost.

In the current work, $NaNO_3$ is used because of its low cost and significantly high latent heat compared to other PCMs that melt at similar temperatures (300 – 400˚C). It is also less hydroscopic (compared to chloride salts/eutectics) which provided an ease in handling. Thermal diffusivity of $NaNO_3$ (industrial grade) was measured using the Linseis xenon flash thermal constant analyzer, XFA 500 that has an accuracy of ± 6%.

The results are presented in Figure 12. The accuracy of the apparatus was verified by a standard graphite sample before and after the measurements.

3.2 Preparation of the PCM Capsules

The PCM capsules or pellets are made in two different geometries namely, spheres and cylinders (Figure 14). The various dimensions of cylindrical pellets are obtained by using respective diameter dies in the power press (Figure 13-a). The spherical shaped pellets of different diameters are obtained from the pelletizer (Figure 13-b) by using water as the binder. Experiments are carried on both kinds of pellets.

The porosity of the pellets is measured with the aid of Sartorius YDK01MS apparatus. For the cylindrical pellets porosity values varied with the force applied in the press and the diameter of the die; specifically it varies with the pressure applied in making the pellet. The porosity of various $NaNO_3$ pellets prepared with the press varied from 3 to 6% depending on the size and forces applied. The porosity of the spherical pellets obtained from the pelletizer varies around 22 to 29%.

The pellets are also made by incorporating an extra void space (between 20-35%) within them (Figure 15). The holes were drilled using drill bits and end mills, and the caps are made in power press. The fit between the cap and the base is transition fit and the void is ensured to be sealed perfectly by using Nickel nanoparticle powder in fitting it.

Advantages and disadvantages of different geometries of PCM encapsulation with different materials and their compatibility are also discussed in literature [7, 9]. Though spherical pellets are best suited for thermal storage applications, cylindrical pellets are still employed as they are easy to manufacture.

3.3 Macroencapsulation of the PCMs

Different techniques have been employed for encapsulating the PCMs. However, developing an economic encapsulation technology using cost-effective materials and processes always remained as the primary notion of the research work implemented. Various approaches followed and the extent of their success are explained and summarized below.

3.3.1 Encapsulation Using Electroless and Electrochemical Coating Techniques

The very first technique which was employed for encapsulating PCM is electroless coating. The idea behind developing this technique is to provide a coating of metal, over the PCM pellets. As the PCM cannot be wetted directly by the metal, and the melting temperatures of the metals are far greater than PCM, dip coating technique did not give better results. So, as an alternate process electroless deposition was attempted.

Sodium nitrate, which was employed in the current study, is dissolvable in water and many other organic solutions. Hence, before carrying out any coating processes, the salt pellets were made impregnable to these solutions by giving a coat with photo polymer Norland 72 (NOA 72). NOA 72 is cured by ultraviolet light between 315 to 400 nanometers and visible light between 400 to 450 nanometers (Figure 16). It can withstand temperatures after aging from -80° C to 90° C. The peak absorption wavelengths are 320, 365 and 420 nm. Full cure requires 5 Joules/sq. cm of energy between 315 and 450 nm. As the NOA 72 is very viscous adhesive, and application over salt pellets is difficult with such viscous liquids, it is diluted with acetone in the ratio 2:1 and the pellets are dip coated. The cure time is ten minutes for the cylindrical pellets with 13mm in diameter. Both the cure and the dilution ratio were determined by trial and error. This curing

process produces a thin film of Poly methyl methacrylate (PMMA) over the $NaNO_3$ salt pellets.

The experiments started with the trials of chemical deposition of metal on the pellets. With a view of depositing Nickel, chemicals like nickel chloride and nickelsulphate hexahydrate were used. Two different solutions were made and are mixed selectively at respective temperatures for obtaining optimum results. The first solution comprised of nickel sulphate hexahydrate (0.171M), nickel chloride (0.063M), boric acid (0.404M) and potassium phosphate monobase (0.33M). The second one had sodium hydroxide (1M) and disodium phosphite hydrated (1M). Both the solutions were taken in equal ratio, and heated to 100˚C. Also few drops of ammonium hydroxide were added upon heating for stabilizing the reactions. It is observed that nickel hydroxide was formed.

In order to optimize the above reaction, few other experiments were also carried out which are described below:

1. Solution 1 and 2 were taken in a ratio of 3:2, and heated to 100˚C and few drops of sodium phosphite (diluted) and sodium hydroxide were added.
2. Solution 1 and 2 were taken in a ratio of 3:2, and a few drops of 1M sodium hydroxide, a few drops of 1M ammonium hydroxide, a few drops of boric acid solution (2gms in 20ml) and a few drops of sodium phosphite solution (1gm in 30ml) were added and the temperature was raised to around 70˚C.
3. To the same ratio of solutions 1 and 2, as stated above, a few drops of sodium citrate solution (5gms per 1 litre), a few drops of sodium acetate solution (5gms per 1 litre), a few drops of thiourea solution (1gm per 1 litre) were added and the temperature was maintained at 85˚C.

The results improved with the modifications made, but still they did not seem to be promising enough. Improvement in the conductivity of the substrate was not significant and also, there was no uniform reaction. Hence, another set of experiments is performed. Three different solutions were prepared. A 60ml solution in DI water (solution A), was made with nickel sulphate hexahydrate (2.63gms), sodium acetate (1.23gms), citric acid (12.61gms) and cobalt sulphate heptahydrate (0.281 gms). A 25ml solution in DI water (solution B) was made with sodium hypophosphite (10.3gms). A 20ml solution in DI water (solution C) was made with sodium hydroxide (9gms) and urea (0.6gms). An electroless coating is performed using the above solutions. In a large beaker 50ml of solution A was taken and while maintaining its temperature at 50˚C, 20ml of solution B was added slowly, later 12.5ml of solution C was added too.

Copper ribbon was left in the solution overnight and a coating of nickel nanoparticles was observed on the top of the ribbon. PMMA coated pellets failed to give the similar results. Some pellets almost dissolved in the solution due to the long stay indicating reactivity of the polymer with it.

During later phases of these experiments, a more conductive coating of Nickel doped polyaniline (PANI) was given on the PMMA coated pellets so that the process of electrochemical deposition becomes easy. The preparation of Ni doped PANI was carried out as described below [53].

The preparation is carried out in two different steps. Firstly, aniline solution, say solution A was prepared by taking 7.31ml of aniline in a burette and adding it drop-wise to the mixture of 300ml HCl (1M) and 1.816gms of nickel nanoparticles present in the beaker at

0˚C and on constant stirring. Secondly, ammonium per disulphate (APS) solution, say solution B, was made by adding 4.56gms of APS in 100ml of HCl (1M). Finally, Ni doped PANI was obtained by adding Solution B drop wise into solution A and leaving it stirring for 12 hours.

Electroless coatings of PANI and graphene (G)-SiO_2 were given to the PMMA coated pellets. By soaking the PMMA coated pellets in the Ni doped PANI solution, polyaniline was in-situ self-assembled over PMMA. Also, the graphene (G)-SiO_2 composite was fabricated using sol-gel technique and the few layers of it were deposited on PMMA coated pellets and SEM image of the composite is shown in Figure 17. It was prepared using TEOS (6.23 grams) and graphene (0.6 grams).

The conductivity of sol-gel G-SiO_2 coated over graphene showed a resistance of .001 ohm and it shows the typical SiO_2 composite structure with graphene. These intermediate coatings improved the conductivity of the substrates further. The electrochemical metal (Nickel and Copper) coating was followed using acid-base reaction and a metal salt as reducing agent. Figure 18 describes the aforementioned hypothetical concept.

The electrochemical technique has been used to coat the metal over PANI or graphene-SiO_2 coated salt pellets [54]. The set-up consists of a cylindrical stainless steel container with a flat bottom acting as an anode, and platinum (Pt) is used a cathode.

In order to achieve a coating efficiently and directly on the salt pellets, the pellets were stirred to form a suspension every 2 minutes by applying 1.2-2.0 V or the oxidation potential of the depositing metal. The presence of conducting pellet could provide an enhancement of mass transport to the cathode in each stirring process. Deposition time is

the critical parameter which determines the coating thickness of the nanoparticles. The resulting metal coated materials were washed with deionized water and methanol, and dried under the vacuum. The formulated procedure failed to give uniform and thick coatings, as the secondary coatings (PANI and G-SiO_2) turned to be porous at the micro scale.

The PMMA reacted with the electrolyte solutions like copper sulphate and the pellets appeared to dissolve in the electrolyte solution during the coating process. Weight loss was observed in the pellets.

3.3.2 Encapsulation Using Silicates

Aqueous solutions of silicates have physical and chemical properties that are useful for coating applications. Thin layers of the silicate solution dries to form a tough, tightly adhering inorganic bonds or films which are non-flammable, resistant to temperatures up to 3000˚F, odorless, non-toxic, moisture resistant, strong and rigid [55].

Water glass is liquid at room temperature and can provide better control of the coating process. Silicate films that are completely dehydrated form a crystalline glassy substance that has a high melting point of about 1800˚F. Typical softening points of 1200˚F and flow points of 1500˚F are seen with films made from high ratio sodium silicates. Sodium silicates are employed in the current study. The potassium silicates are similar to sodium silicate but have properties that are better suited to some applications, e.g., when greater electrical resistance is required [55].

Silicates are converted to solid films or bonds by two methods: evaporation of water (dehydration) or chemical setting mechanism [55]. Evaporative drying is a process where

water evaporates making liquid silicates turn progressively tackier and more viscous is implemented over plain salt substrates and polymer coated salt ($NaNO_3$) substrates. Curing of sodium silicate involves applying a very thin layer of sodium silicate over the pellets and increasing the temperature slowly to 200-210˚F to slowly remove water and prevent bubbling of the film. Final curing can be done at 300-400˚F. Three different types of liquid sodium silicates, N, K, RU were employed in the current work. The $NaNO_3$ pellets coated with these three types of silicates Figure 19(a) and Figure 19(b) show surface defects after cure. Since the salt pellets absorbed the water from these silicates and as the water escapes from the pellets at higher temperatures, it gave rise to bubbles in the coating. Apart from bubbling, blisters were also observed on the $NaNO_3$ pellets even when curing was performed in a vacuum furnace at very low heating rates. Adding to these defects, curing using this evaporation process consumed very long time as rapid heating causes the film to blister by turning water in the film to steam. Whitening of the coating, also called as efflorescence or blooming, is another defect which was observed and occurs due to reaction of carbon dioxide in atmosphere with the sodium in the sodium silicate coating to form sodium carbonate on the surface of the coating. Efflorescence occurs especially in hot, humid conditions. Inspite of carrying the curing process in the vacuum furnace this defect was noticed.

Another process adopted was to use additives in sodium silicate. When TiO_2 is used as the additive [56], the defects got minimized to a large extent over glass substrates but no significant improvement was observed on salt substrates. Attempts were also made for curing the silicate coatings in the microwave at low powers, but when the temperature of the cured pellets was raised to 200˚C, the surface defects repeated again.

In conclusion, the application of sodium silicate coatings through evaporative drying technique is not preferred as the salts absorb the water from liquid sodium silicate.

The chemical setting method, where an alkali silicate is neutralized with acidic materials to polymerize the silica and form a gel, can be opted in this situation. Chemical setting is often used to improve film moisture resistance, to reduce setting time, and to increase ultimate bond strength as needed. Multivalent metal compounds react with silicate solutions to form coatings, leaving behind the insoluble metal silicate compounds as precipitates [55]. However, the feasibility and the extent of applicability of this method need to be investigated further.

3.3.3 Encapsulation Using Sand

Owing to the advantages like low cost, low degradability, high strength and almost negligible reactivity to metal oxides and molten salts, sand encapsulation was found interesting. However, it has disadvantages like porosity, moisture absorbability, etc. However, by employing proper techniques these shortcomings can be overcome and this can be used as a potential concept for thermal energy storage. The research work done in applying this concept along with the results is presented below.

Development of Composite of Sand and PCM: The spherical $NaNO_3$ pellets prepared in the pelletizer are dried in the oven at 100℃, for 24 hours to get rid of moisture in the salt. Silanization process is followed; the pellets are immersed in the hexane (20ml) and silane (200µl) for 24 hours. The pellets are heated at 200˚C for 1 hour so that the excess liquid evaporates and the pellets get dry. The pellets are then coated with a polymer, flash heated at 250˚C and the outer surface is dried, and then they are cured till 250˚C at a rate

of 4˚C /min (Figure 20 a). Then sand with sodium silicate as binder (5% wt to wt ratio) is taken. The pellet is then encapsulated in the sand, by power press with a force of 10 tons for two minutes. It is then microwaved for ten seconds at 80Watts (10% power of the microwave) power [57] and heated at 200˚C so as to ensure that moisture escapes. Finally these sand encapsulated pellets are immersed in hexane and silane solution for salinization (Figure 20 b). The pellets are then heated to 340˚C and left for three hours and later cooled to room temperature (Figure 20 c), and the cross section was observed (Figure 20 d). The molten salt diffused into the pores of the sand and hence, there was no leakage observed.

The particle distribution also plays an important role in obtaining highly dense non-porous shells. The packing density is kept to maximum by taking selective sand sizes [58].

On the whole, application of this technology needs further analysis.

3.3.4 Encapsulation Using High Temperature Resins

Further advancing and application of new ideas required. Hence further work was done by adopting high temperature resistant polymers.

High temperature resins like bisphenol A diglycidyl ether with epoxy embedding medium hardener MNA, polybenzimidazole (PBI), and also few kinds of polymers were studied for the encapsulation over PCM (sodium nitrate in the current study).

The diglycidyl based resins used with epoxy hardeners can withstand several thermal shock cycles between -65˚C and 150˚C [59] in a dry environment. Also, these resins failed in wetting the salt substrates. The resins were wet even after curing. Though they

are cost effective and possess good mechanical and thermal characteristics, they are not compatible with the PCMs and thus discarded.

The polybenzimidazole (PBI) is another high temperature resin which can withstand temperatures upto 400 ˚C. The resin used was 26% PBI solution containing dimethyl acetamide (DMAc). Dimethyl acetamide (DMAc) aids in reducing the viscosity and increases the ease of handling of the resin. Besides high thermal stability, PBI possesses high chemical resistance [60] to most organic chemical systems. Even at elevated temperatures, effect on mechanical properties or dimensional properties was negligible. Yet, compatibility of this polymer with the PCM turned out to be very limited (Figure 21). After the cure (cure process suggested by PBI Performance Products, Inc.), the PBI coating got peeled away. Attempts of multiple coatings did not help as well.

Additional high temperature polymers were considered, and one of them was found to be suitable. Figure 22 shows polymer coated pellets and Figure 23 shows polymer and Nickel particle coated and cured pellets.

The concept of encapsulation of PCMs with a metal oxide (SiO_2) contributes in achieving high temperature performance (300˚C – 500˚C) with cyclic performance capability. Many experiments were done using different ratios of chemicals like silane and alcohols, and finally the reaction for SiO_2 deposition was optimized. Initially the polymer coated salt capsules were pre-heated to 200˚C over a hot plate. A solution of tetraethyl orthosilicate (TEOS) and 0.25M HCl were mixed in ratio 5:1, and added to the polymer coated capsules in such a way that the solution wets the entire surface area. Later, the temperature of the capsules was raised to 250˚C and (3-Aminopropyl)triethoxysilane was

added over the capsules. These reactions of self-assembly, hydrolysis and simultaneous chemical oxidation occurring over the capsules at different temperatures produce the metal oxide coating. Figure 24 shows an encapsulated pellet. Yet implementation of this concept comes with many obstacles which have to be addressed in order to implement the process successfully at a large scale.

Few pellets prepared using above encapsulation concept gave credible results, but the consistency and the repeatability of the process needed to be stabilized and optimized. Factors which affect the encapsulation process were identified and troubleshooting methods are being developed to achieve perfect encapsulation of the PCM pellets.

3.4 Considerations in Encapsulation Process Using High Temperature Polymers

The concept of encapsulation of PCMs with a metal oxide (SiO_2) contributes in achieving high temperature performance (300 – 500˚C) with cyclic performance capability. Yet, the implementation of the above concept has encountered many difficulties which have to be addressed in order to implement the process successfully at a large scale.

3.4.1 Provision for Expansion, Void Space

The most important factor which affects the effectiveness of the encapsulation is the void space for thermal expansion of PCM. Since $NaNO_3$ pellets obtained from the power press possess porosity of only 3 to 6% (depending on the force applied and the dimensions of the pellet), while the volume change of sodium nitrate from the solid phase to the liquid phase is reported to be about 10.7% [61], it is necessary to prepare the $NaNO_3$ pellets with enough void space to take care of the expected volume change. Hence, pellets were fabricated, by providing space for thermal expansion. The fabrication is accomplished by

milling the pressed pellets. The developed conceptual design of the void and the pellets prepared with void are shown in the Figure 25. These pellets, prepared by joining these two parts, were 30mm diameter and 12mm height and had a void space of about 34% to take care of the volume change of the PCM.

3.4.2 Surface Characteristics of the Materials

The SiO_2 coatings at times turned out to be highly non uniform. The thickness values of SiO_2 coating, when observed under Leitz optical microscope, for a sample pellet (Figure 26) varied from 0 to 75 micrometers. Increasing the surface roughness of the substrates has helped in providing more uniform coatings.

3.4.3 Uniformity of Coatings

The first concern is to achieve uniform coatings. Dip coating of the precursor solution is not a good option as the extra solution over the $NaNO_3$ pellets would stick out after cure. During the curing process, the viscosity of the solution reduces as the temperature increases and excess solution drips down the $NaNO_3$ pellets. Hence a thorough application of the right amount of precursor solution is required. At the lab scale the solution is applied over the pellets by hand or brush. The method of application is still being optimized to implement at a large scale.

After performing various experiments one of the methods produced better results and is described as follows. As the heat tests were carried out on the coated pellets, the problem came up from the high pressure which was created by the air in the void of the capsule. Evacuation of the void turned out to be difficult. One of the ideas developed to address this problem was to prepare a capsule with a void. A hole was drilled and same diameter

wire was inserted in it (Figure 27 a). The polymer is coated and cured (Figure 27 b, c), followed by giving the pellet SiO_2 coating (Figure 27 d) and the capsule was then heated to 200˚C and the heated air was allowed to escape from the void through the pin-hole. The pin-hole was sealed while the pellet was hot (Figure 27 e). This enabled the air to escape at elevated temperature and hence reduce the pressure within the capsule. When the pellet was subjected to thermal cycling, it survived for 7 cycles (Figure 27 f) before leakage was observed. The leakage observed in the eighth cycle is shown in Figure 28. This showed that the lower air pressure in the capsule helped in achieving a greater number of cycles before the failure occurred. Upon extended cycling a reaction might have occurred between salt and the polymer which resulted in reducing the strength of the encapsulating coating by making it porous which eventually lead to leakage of salt. Further research is required to optimize this encapsulation process.

3.5 Characterization

In order to prepare a hard metal oxide coating over the pellets, a precursor solution is coated over $NaNO_3$ pellets. The reactivity of the solution with the molten $NaNO_3$ plays an important role, keeping in mind the sustainability of the $NaNO_3$ capsules over a large number of heating and cooling cycles. The reactivity of the solution with the $NaNO_3$ is studied by characterizing the coatings using FTIR and SEM on different samples.

3.5.1 Fourier Transform Infra-Red Spectroscopy (FTIR)

Pellets of potassium bromide (KBr) are used as KBr is transparent to IR radiation. A sample of KBr pellet was coated with the precursor solution and cured by heating to 250˚C and 300˚C. The FTIR analysis indicated the absence of reaction between the KBr

and the coating, up to temperatures of 300˚C. The test was repeated with $NaNO_3$ and the coating material on a glass slide which confirmed that there is no reaction between the coating solutions and $NaNO_3$.

3.5.2 Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX) Analysis

Three samples of $NaNO_3$ pellets with 34% void space and coated with multiple layers of precursor are cured by heating the samples to 250˚C at the rate of 4˚C per minute. After the samples are prepared, they are heated to 350˚C. The EDX and SEM results are presented in Figure 29 and Figure 30.

EDX results are shown in Figure 29 and they show the presence of only Na, N, O and C suggesting that no other element is present. The elemental composition of the coating interface is shown in Figure 30. No other elemental composition is observed except the $NaNO_3$ and precursor coating materials.

However, these tests show that the intermediate polymer coatings and the molten salts don't react during the test period. Sustainability of this concept under extended cycling still needs to be studied. Based on the characterization analysis performed till now, it is anticipated that the polymer coatings will not react with the salt and the concept can be successfully implemented.

3.6 Thermal Cycling

The performance and efficiency of the encapsulation coatings is verified through heating and cooling cycles. The cyclic process that is used for testing the encapsulated pellets in the furnace is shown in Figure 31.

Some of the $NaNO_3$ capsules prepared using the above encapsulation method were able to withstand up to 7 heating and cooling cycles. The consistency and repeatability of the process needs to be stabilized and optimized. Efforts are continuing to optimize the technique of macroencapsulation.

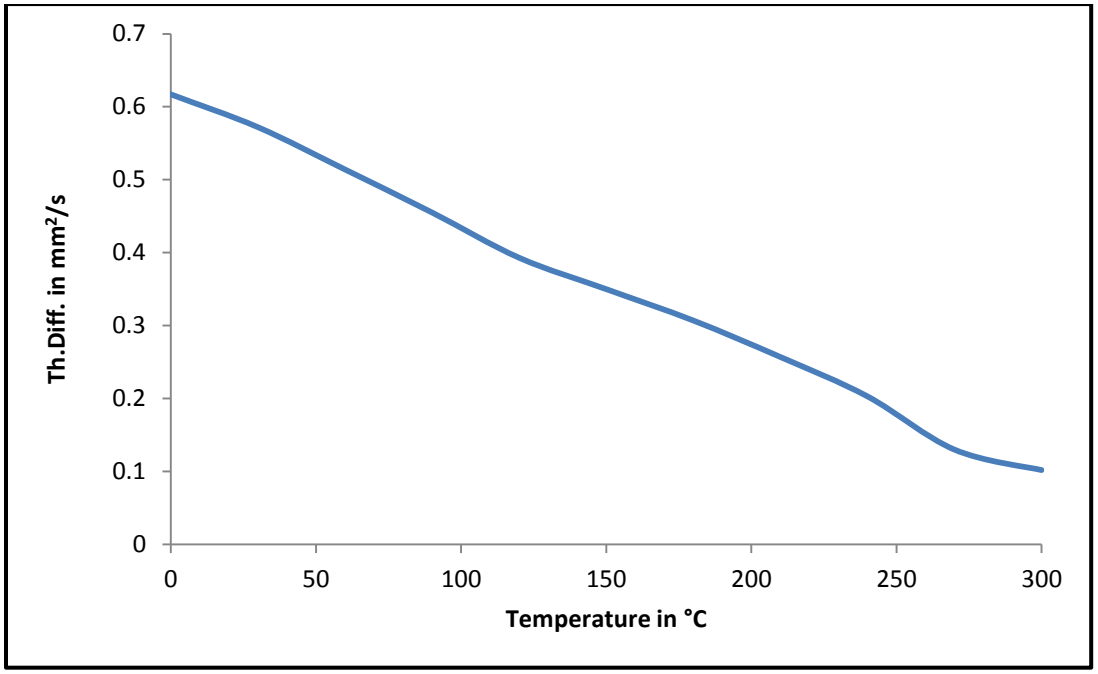

Figure 12: Thermal diffusivity of $NaNO_3$

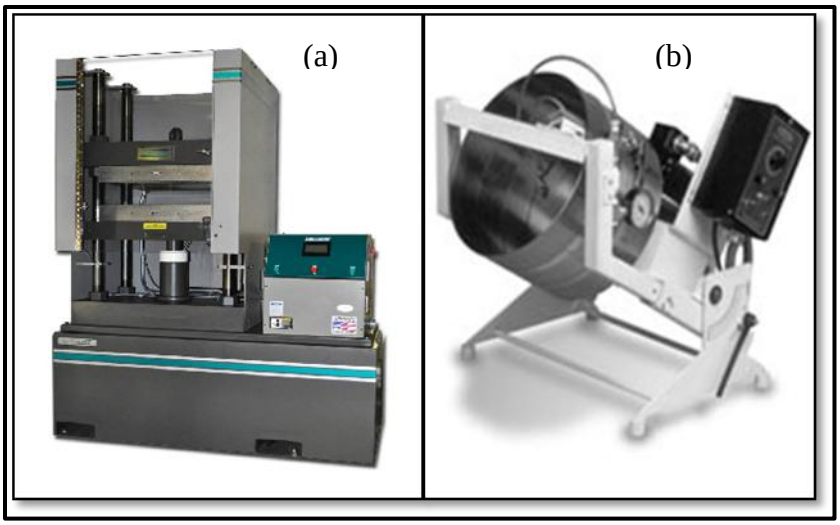

Figure 13: Lab scale equipment: (a) power press (b) pelletizer

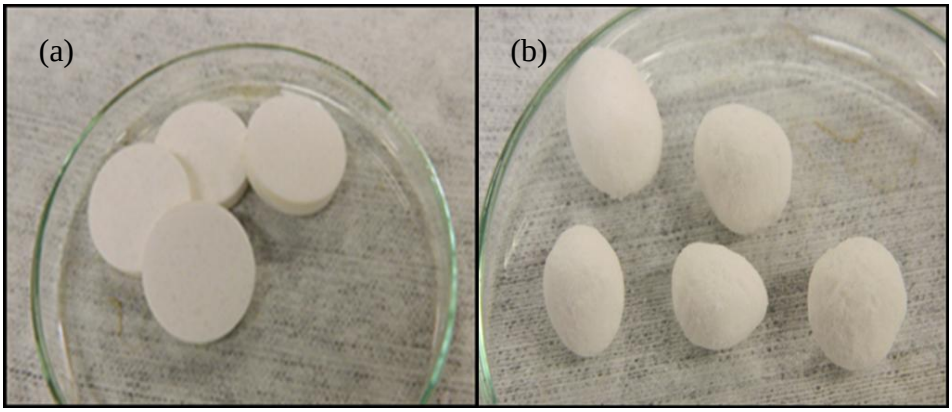

Figure 14: Pellets prepared from (a) power press (b) pelletizer

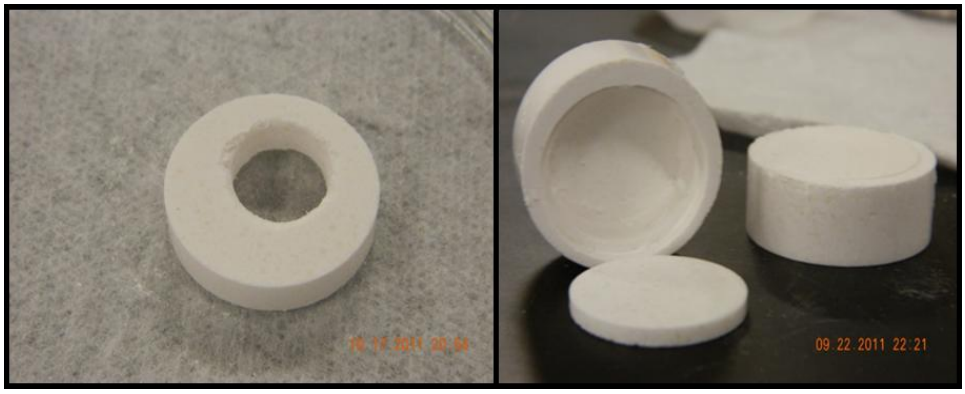

Figure 15: The pellets with void space

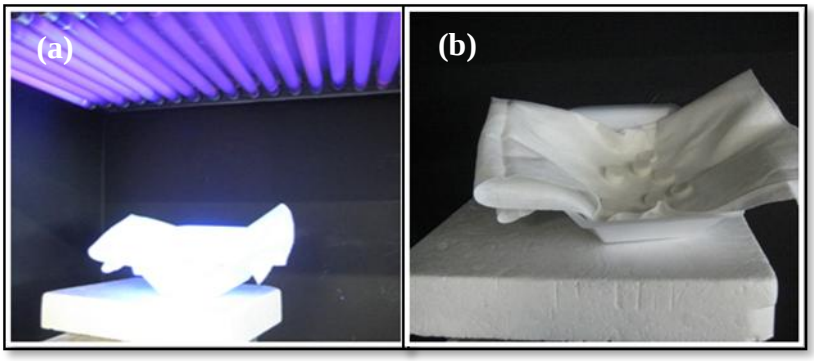

Figure 16: Coating of NOA (a) pellets under UV light (b) cured pellets

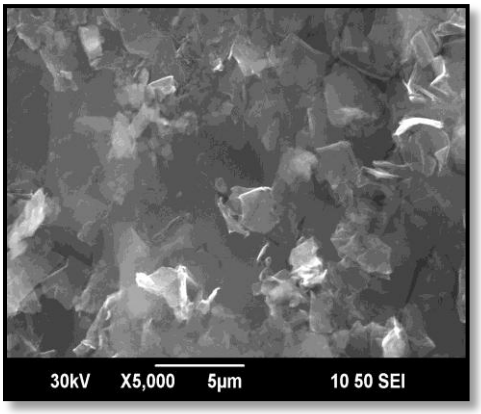

Figure 17: Sol-gel of G-SiO_2

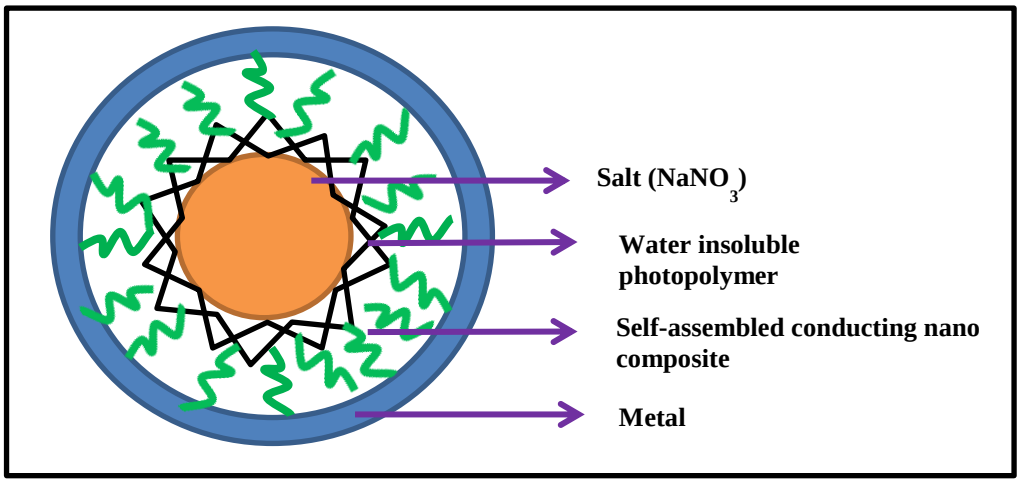

Figure 18: Hypothesized schematic of the encapsulated pellet

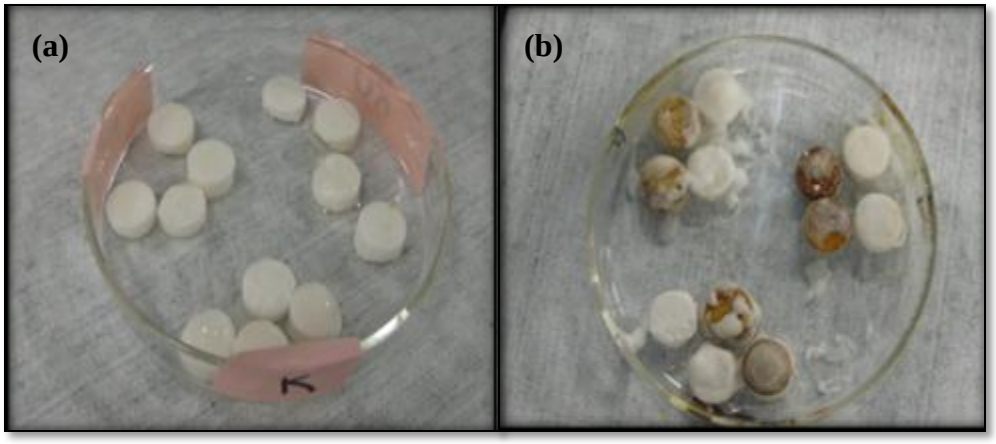

Figure 19: Pellets with sodium silicate (N, K, RU types) coating

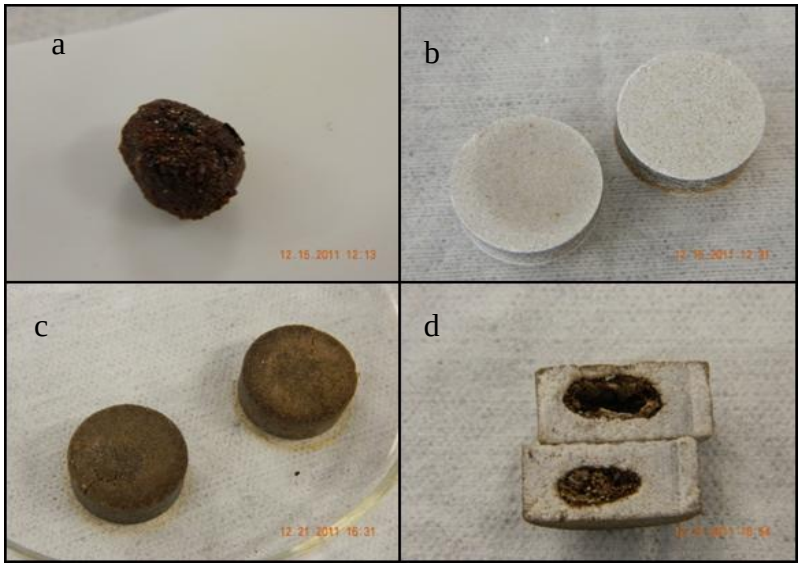

Figure 20: Encapsulation of salt in sand

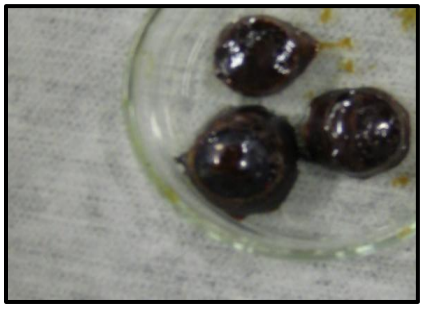

Figure 21: The PBI coated pellets

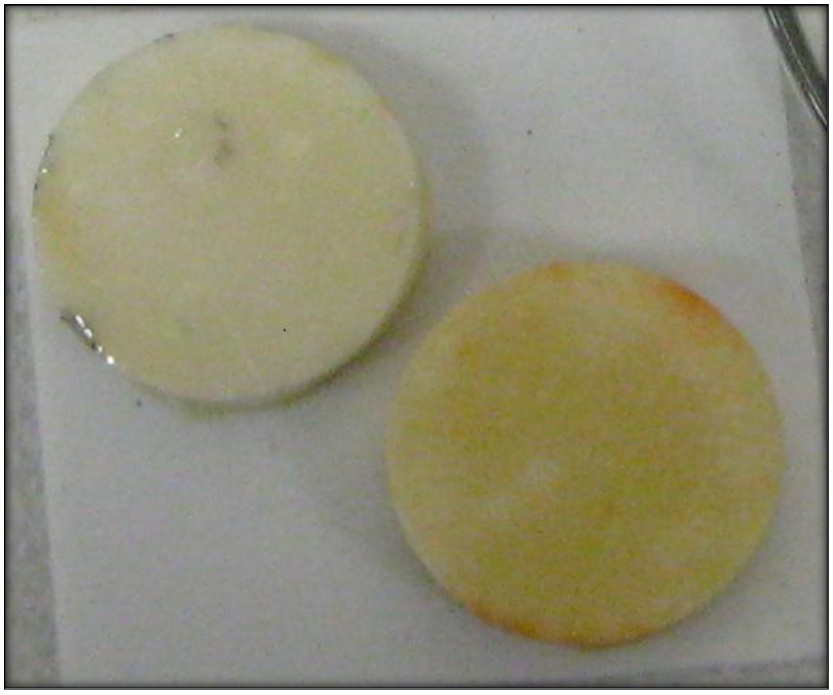

Figure 22: Polymer coated pellets

Figure 23: Polymer and Ni-polymer coated and cured pellets

Figure 24: The SiO_2 coated over $NaNO_3$ pellet

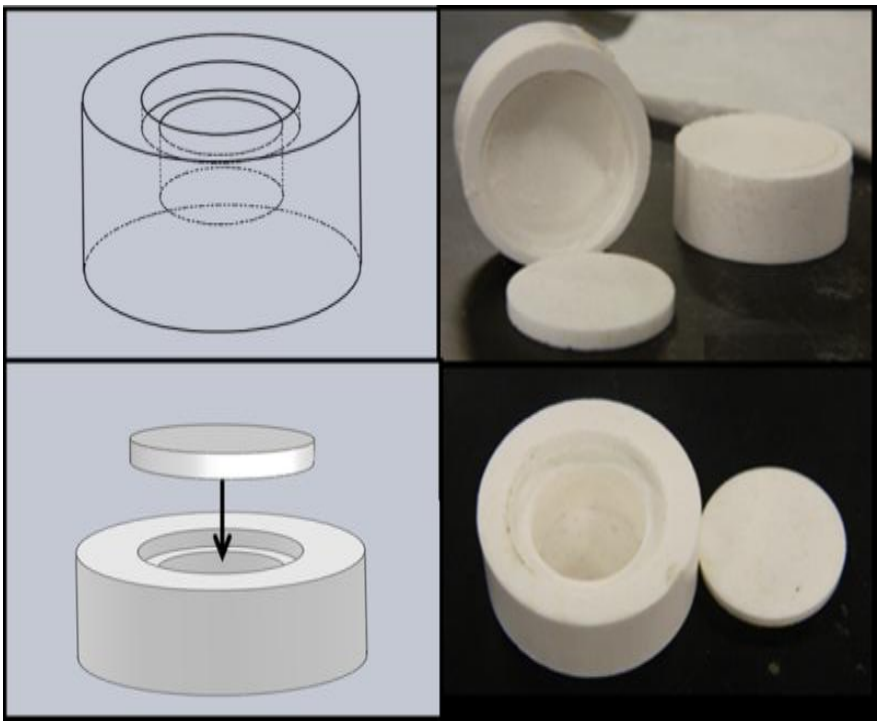

Figure 25: Conceptual design of void and pellets prepared with void

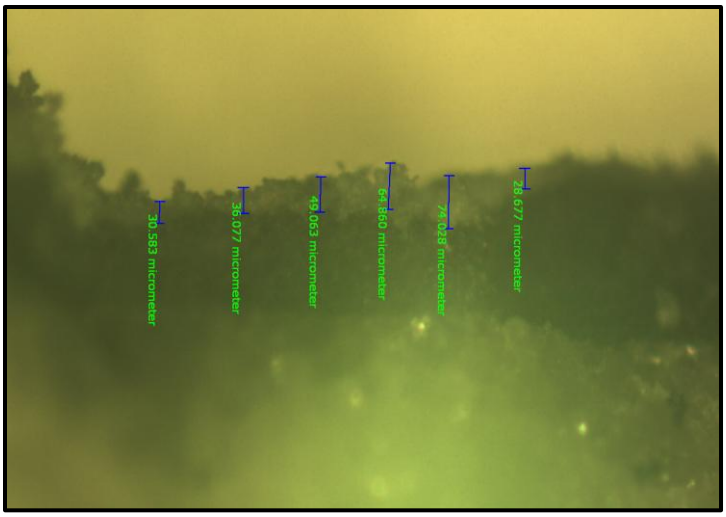

Figure 26: Thickness of the SiO_2 layer

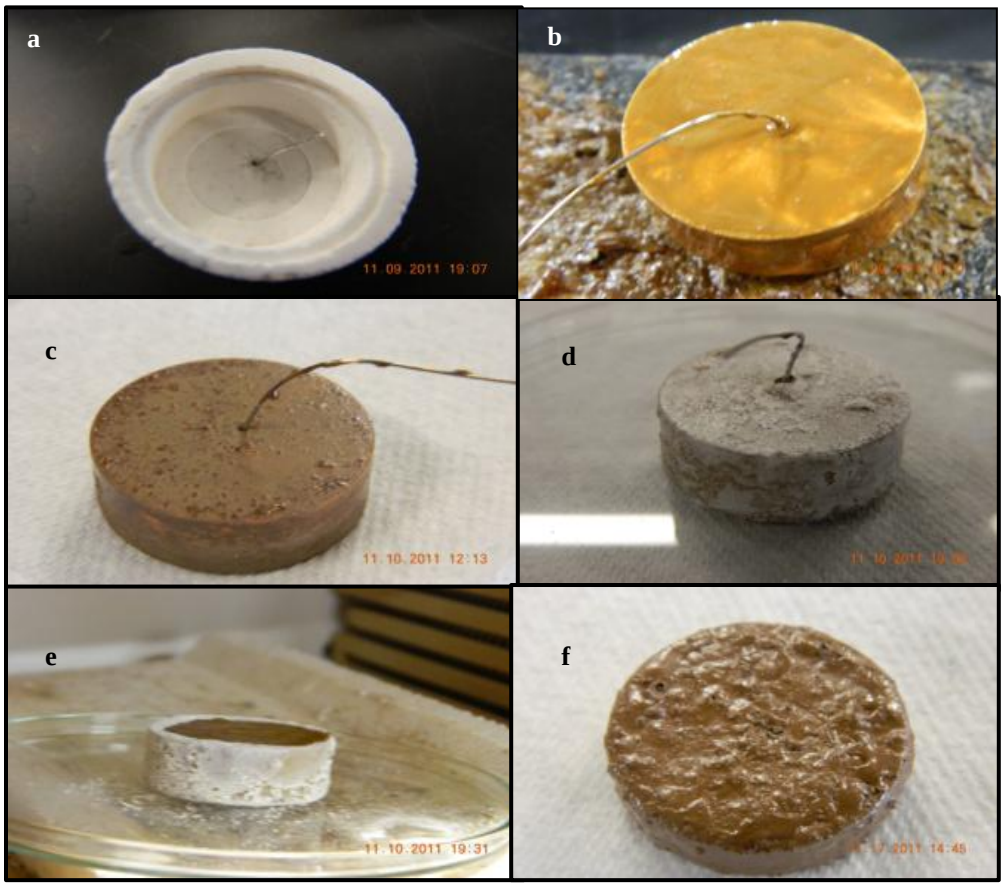

Figure 27: Preparation of the pellet

Figure 28: The pellet showing salt leakage

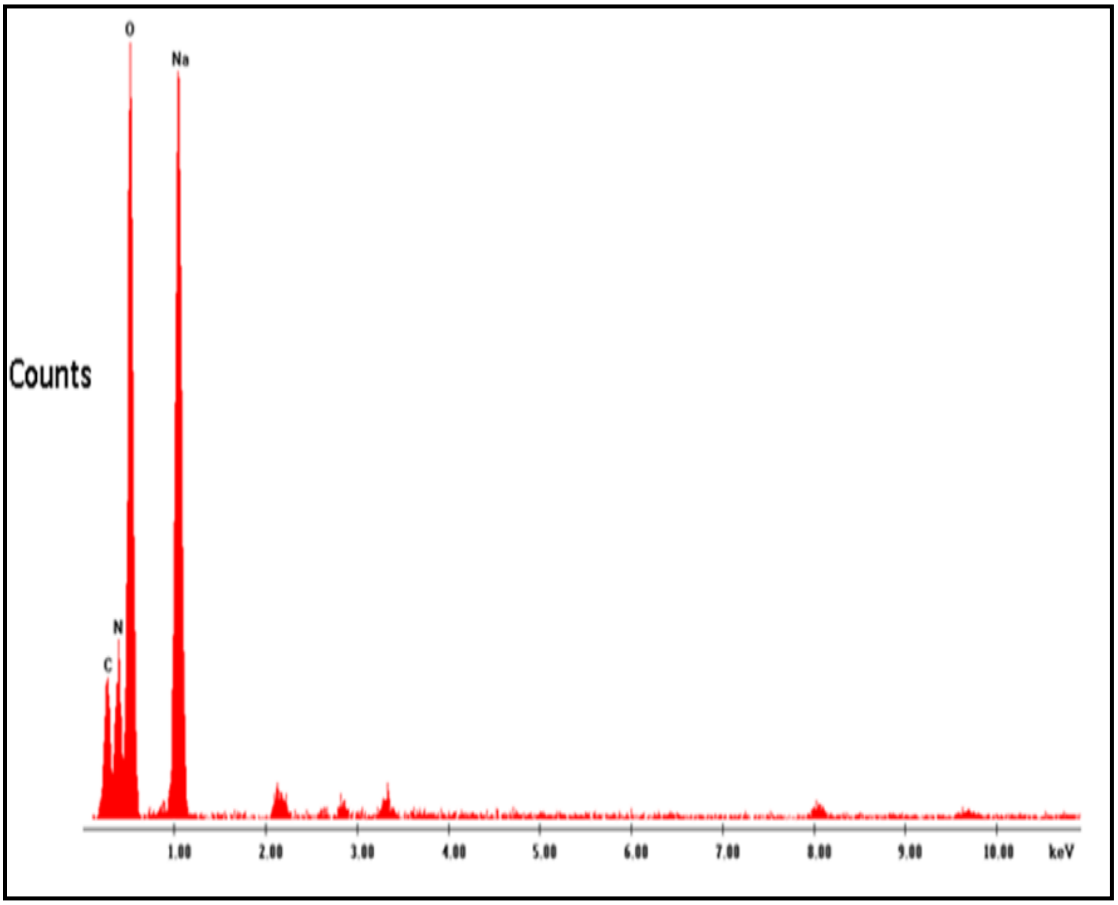

Figure 29: EDX of $NaNO_3$ with 3 layers of precursor coating

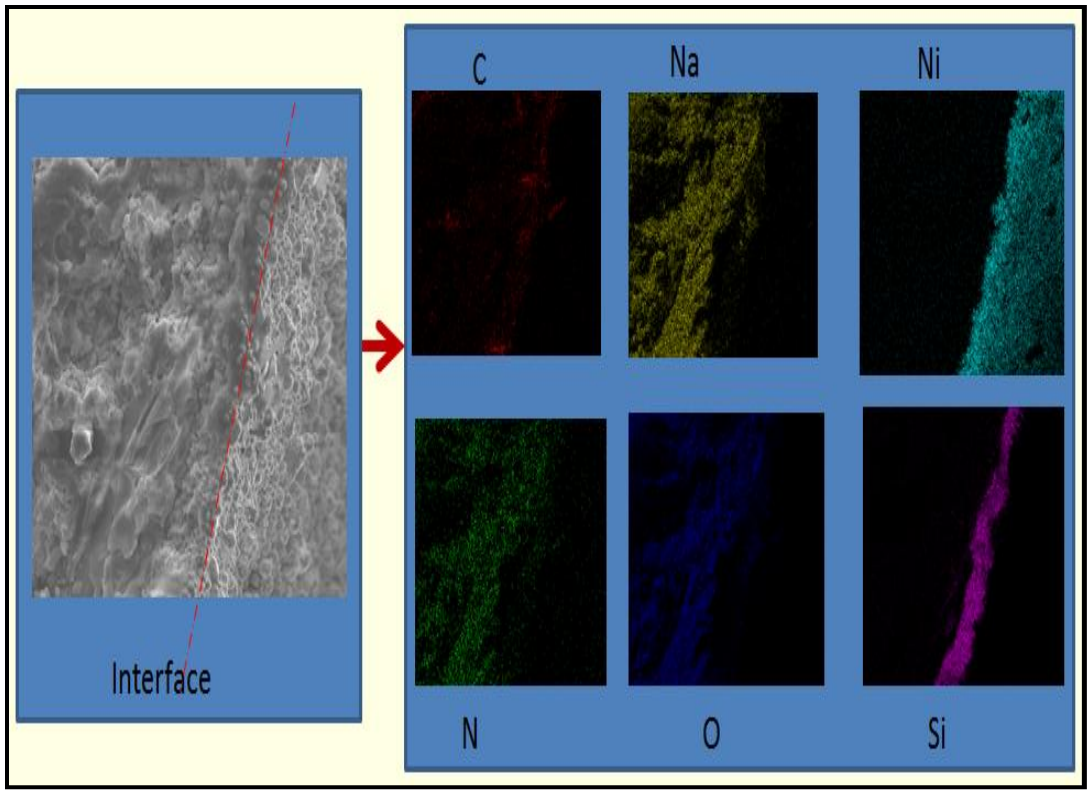

Figure 30: SEM analysis

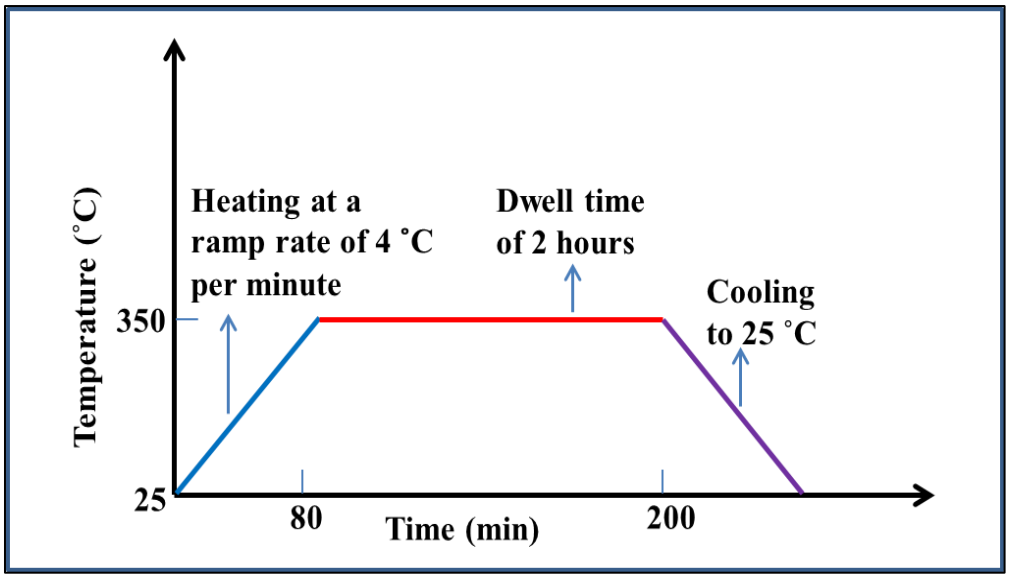

Figure 31: The heating and cooling cycles

CHAPTER 4: SUMMARY, CONCLUSION AND RECOMMENDATIONS

4.1 Summary

The quest for new technologies to avert the growing concern about environmental problems, the imminent energy shortage and the high cost of energy and new power plants have been a scientific concern over the last three decades. Central to the problem is the need to store excess energy that would otherwise be wasted and also to bridge the gap between energy generation and consumption. Latent heat thermal energy storage is particularly attractive technique because it provides a high energy storage density. When compared to conventional sensible heat energy storage systems, latent heat energy storage system requires a smaller weight and volume of material for a given amount of energy. In addition latent heat storage has the capacity to store heat of fusion at a constant or near constant temperature which correspond to the phase transition temperature of the PCM. In order to improve the energy storage efficiency and reduce costs, proper design of the system is necessary. Encapsulation of PCMs increases the efficiency of the TES, by resolving the problem of low thermal conductivity upto an effective extent. Encapsulation of low cost PCM with a metal oxide (SiO_2) for a storage system in a temperature range of 300˚C - 500˚C has been developed. These coatings have been characterized and cyclic stability, thermal performance of the capsules is analyzed.

The extent of applicability of concepts of encapsulation like electroless and electrochemical coatings, coating using silicates, sand encapsulation has been studied.

The potentials realized behind considering these concepts and hardships faced in implementing them were elaborated. The pros and cons were discussed in detail.

4.2 Conclusions

Encapsulation of low cost PCMs such as $NaNO_3$ with a metal oxide (SiO_2) for a storage system in a temperature range of 300˚C - 500˚C has been developed. First, a high temperature polymer, is coated over PCM pellets, and cured, so that the pellet becomes insoluble in water as well as several organic solvents. Then the metal oxide is coated over the pellet using self-assembly, hydrolysis, and simultaneous chemical oxidation at various temperatures. The understanding of the process, extent of accomplishment and the preliminary characterization results were reported.

Apart, from the above technology, other potential concepts were also explored. The shortcomings which led to failure in the execution of the electroless and electrochemical coatings were discussed. The application of silicates (water glass) has been studied and the practical difficulties in implementing it were described. The novel idea of sand encapsulation has been introduced and primary results obtained in applying this concept were discussed.

4.3 Recommendations

A novel technology has been developed is to encapsulate lost cost PCMs with metal oxides. A high temperature polymer is coated over the PCM initially and then metal oxide is coated over using self-assembly, hydrolysis, and simultaneous chemical oxidation at various temperatures. The performance and the cyclic stability of these metal oxide encapsulated pellets can be improved by developing the quality of the polymer

coatings. Also, the reactivity of the polymer with the molten salts upon extended cycling on long run should be studied.

Another potential concept which can be investigated in future is the application of silicate coatings using chemical setting process. The application of evaporation drying method in case of salts couldn't yield satisfactory results as the salts absorb water from the silicates which is very difficult to remove. In chemical setting process multivalent metal compounds react with silicate solutions to form coatings by precipitation of insoluble metal silicate compounds and it provides extra advantages like improving film moisture resistance, reducing setting time, and increasing ultimate bond strength.

Incorporating sand as encapsulating materials has a unique significance as well. The sand combined with liquid sodium silicate as a binder and cured can be used for encapsulation. The strength and chemical resistance improve longevity the sand shells. By handling the problems of moisture absorbability and porosity by adopting the proper designs and curing methods this technology may be promising approach for TES.

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APPENDIX A: NOMENCLATURE

$Q_{sensible}$ = sensible heat stored in the material

m = mass of the material in which heat is stored

C_p = specific heat

T_1 , T_2 = Temperatures ($T_1 < T_2$)

Q_{latent} = latent heat stored in the material

T_m = phase change temperature ($T_1 < T_m < T_2$)

L = heat of phase transformation

$Q_{chemical}$ = heat stored in the material during a chemical reaction

a_r = fraction reacted

Δh_r = heat of reaction per unit mass

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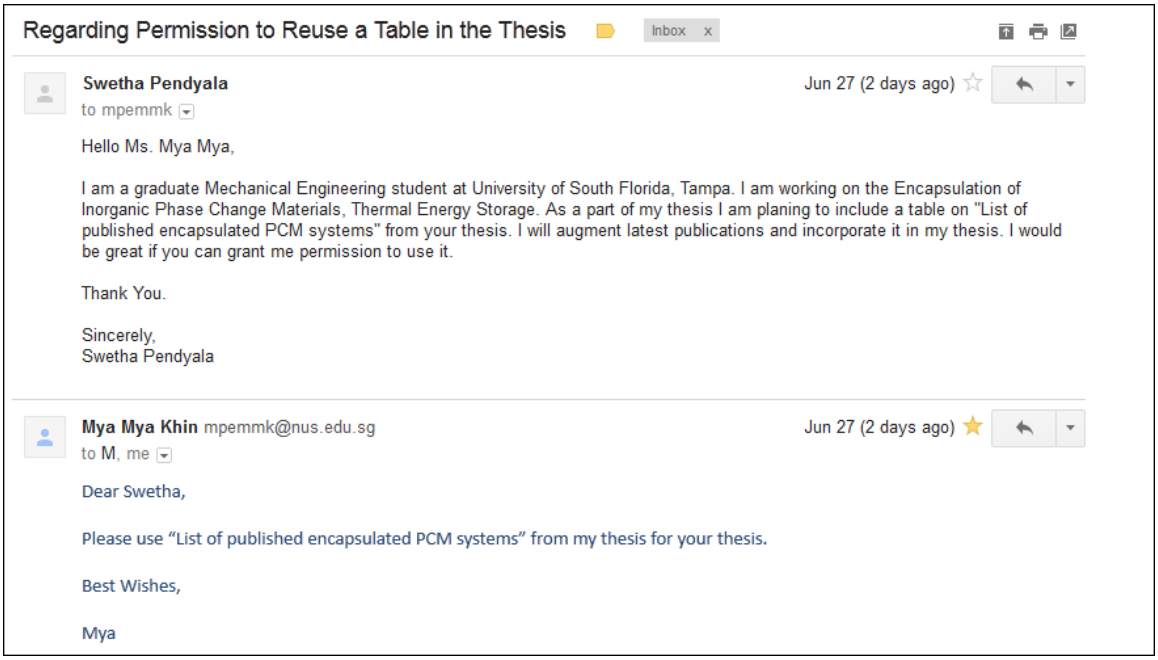

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Swetha Pendyala Jun 27 (2 days ago)
to mpemmk

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