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Encapsulated high temperature PCM as active filler material in a thermocline-based thermal storage system

B. Muñoz-Sánchez^a, I. Iparraguirre-Torres^{a*}, V. Madina-Arrese^a, U. Izagirre-Etxeberria^b,
A. Unzurrunzaga-Iturbe^b and A. García-Romero^c

^aSolar Energy Unit. Tecnalia Research and Innovation, Mikeletegi Pasealekua, 2 - 20009 San Sebastián (Gipúzcoa).Spain.

^bMaterials for Energy and Environment Unit. Tecnalia Research and Innovation, Mikeletegi Pasealekua, 2 - 20009 San Sebastián (Gipúzcoa).Spain.

^cDpto. Ing. Minera-Metalúrgica y C. Materiales, Universidad del País Vasco (UPV/EHU), Rafael Moreno "Pitxitxi", 2 – 48013-Bilbao (Vizcaya). Spain.

Abstract

A great concern in Concentrated Solar Power (CSP) is to boost energy harvesting systems, by finding materials with enhanced thermal performance. Phase Change Materials (PCM) have emerged as a promising option, due to their high thermal storage density compared to sensible storage materials currently used in CSP. A thermal storage system for solar power plants is proposed, a thermocline tank with PCM capsules together with filler materials, based on multi-layered solid-PCM (MLSPCM) thermocline-like storage tank concept [1,2]. A detailed selection of the most suitable high temperature PCM, their containment materials and encapsulation methods are shown.

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* Corresponding author. Tel.: +34 946 430 850.
E-mail address: inigo.iparraguirre@tecnalia.com

1. Introduction

Feasibility of solar thermal technology has been widely demonstrated, becoming a safe bet in the energy mix. Growing interest from both public and private sector has led to achieve considerable advances and improvements in recent years.

However, one of the critical factors to make this energy option more competitive is to extend plant operation time when sun is not available (cloudy weather or at night). This would allow to produce energy in a constant way and to adapt operation to energy demand.

Current commercial CSP plants have implemented solutions to resolve the aforementioned issue. The first option is called hybridization, which implies burning natural gas or biomass to supply heat to the system, increasing carbon footprint of a renewable energy as solar is. This situation generates the need for an effective method through excess heat could be saved for later use. Thus, second possibility consists of storing thermal energy when sun is shining to use it afterwards.

Thermal energy storage (TES) is a wide technology field which has been long studied in different industries and processes. Recent rise of solar thermal power in the last decade has led both industrial and academics to prove its technical viability in solar power plants. Although these industrial facilities are more and more being installed with reliable and robust mechanisms to store thermal energy, they still can be improved to gain efficiency and reduce costs.

In this sense, the two-tank molten salt system is the most widespread and commonly employed to accumulate thermal energy. This work proposes an alternative to the mentioned system based on a single tank filled with heat accumulating materials.

2. Conceptual design

A TES system encompasses three main concepts: the storage medium, heat transfer mechanism and containment system. The storage medium refers to the material which loads thermal energy. Its nature and characteristics highly depends on heat storage mechanism, involving a change in material internal energy. This can be produced in three ways, the first two being the most widely used in TES systems; sensible heat, latent heat and thermochemical storage [3-8].

The most of recently built CSP plants have an active indirect sensible storage system with molten salts as storage media. The advantages of the two tanks indirect system are that cold and hot material are stored separately and they flow only between these two tanks not through the parabolic troughs. As main disadvantage, the salt mixture freezes at very high temperatures, around 220 °C depending on composition. This means higher maintenance costs in order to avoid salt solidification. In addition, the storage material itself has high costs because extensive volumes are needed in order to achieve enough stored energy and required heat exchangers are also expensive.

On the other hand, latent heat of fusion is much greater than the specific heat for any substance. That makes PCM to have larger volumetric energy storage capacity than sensible storage materials, with subsequent smaller storage sizes.

Though PCM storage has been experimented for solar plant applications, it has not been employed in commercial facilities yet. The development of high temperature thermal storage using latent heat is an area of increasing interest due to the mentioned advantages. However, most of PCM have a low thermal conductivity, leading to low charging and discharging rates and sometimes unsuitable range of temperature in the material.

The solution proposed in this work consists of the use of encapsulated PCM to enhance the thermal properties of the melt salts used in sensible thermal storage systems.

2.1. Thermocline system

An alternative to the mentioned two tank of molten salts is the single tank thermocline system [6,7,9,10]. Hot and cold fluids are placed in the same tank and they are separated due to thermal stratification. Thermal storage material can be partially replaced by a low cost filler material (sand or rocks), which has similar thermal capacity and it is

cheaper than molten salts. However, it is necessary to carefully control charging and discharging processes to maintain the thermal gradient inside the tank.

One significant improvement over this system can be achieved by replacing part of the filler material by PCM. The idea is to place several PCM with different melting points inside the thermocline tank in such a way that the highest melting point material is at the top and the lowest stand at the bottom of the tank. The heat in the thermocline tank is stored by the melt salt, the filler material and also by the encapsulated PCM. In this case, quoted material will be a PCM, storing thermal energy in form of latent heat. Ideally, various PCM should be placed along the tank to achieve a more efficient and stable system. This configuration could allow near isothermal heat transfer process, resulting in a more efficient system from a thermodynamically point of view.

The storage tank system using encapsulated PCM and sensible heat transfer fluid (HTF) has been still previously proposed either for low temperature [11] but also for CSP [12], showing a good performance through simulations.

A recent publication [13] shows the simulation of tank with a cascaded filler structure composed of multiple encapsulated PCM with their melting temperatures tuned along the tank height. It has been concluded that modification of the porous bed with a cascaded PCM structure increases the utilization of the latent heat and can provide significant improvement over conventional quartzite rock filler.

The present paper analyzes a system based on the novel multi-layered solid-PCM (MLSPCM) thermocline-like storage tank concept proposed by Galione et al. [1,2]. These authors have shown that this system is a promising alternative to two-tank and standard thermocline TES.

The main contribution of this research lies in the development of real materials and techniques which are capable to be an active part of the system, whereas mentioned existing literature has been focused on obtaining results based only on simulation techniques.

The intended thermal storage system is considered for both, parabolic trough with thermal oil as heat transfer fluid (HTF) and central tower with melt salts as working fluid. In both cases, a mixture of nitrates containing 60% NaNO_3 and 40% KNO_3 (solar salt) is used.

2.2. PCM

Latent storage materials integrated in the TES system should be chosen under several constraints to maximize the amount of energy stored and to obtain the best efficiency. Melting temperatures, encapsulation details (size, shape, materials and storage capacity) and costs are factors to be considered.

Regarding melting point temperatures, different PCM will be chosen to match different HTF temperatures. Ideally, various PCM should be placed along the tank to achieve a more efficient and stable system. Thus, three target melting temperature ranges have been defined, related to income and outcome molten salt temperatures.

In this sense, maximum temperatures achieved by HTF in solar power plants depend on capture technologies. For parabolic trough plants with thermal oil, HTF comes from solar array at maximum temperature of 395 °C while in central tower with solar salt higher values are reached, about 565 °C. Being aware of this, when selecting the PCM for the upper part of the thermocline tank, melting temperatures of around 5 to 25°C less than the HTF maximum temperatures are required to guaranty a good performance and phase changing of the PCM. These temperature ranges have been established along with specialized bibliography recommendations [14]. Thus, the required PCM for the upper part of the tank are of 370- 390°C for the parabolic trough system and 540-560°C for the tower configuration.

For the definition of the required melting temperature of the PCM to be located in the lower part of the thermocline, for both cases (parabolic trough and tower) the same value is used. In both cases, the cold salt is stored at around 290°C to guaranty that it is maintained liquid. With this in mind, the PCM needs to have a melting temperature of about 5 to 25°C higher than 290°C (melt salt inlet temperature).

Figure 1 represents the proposed TES system and melting temperature ranges for each PCM.

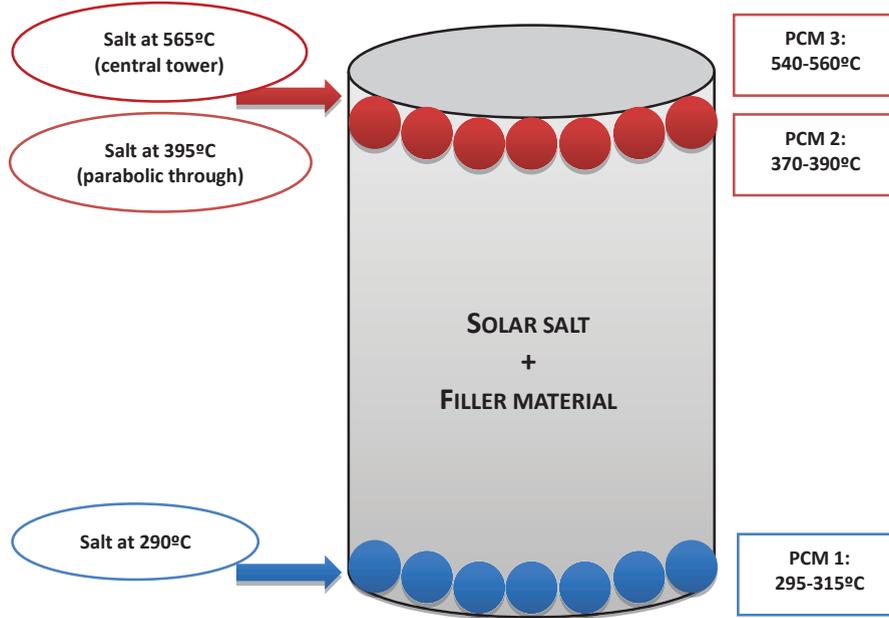


Fig. 1. Thermocline tank and PCM melting temperature ranges of proposed configuration.

2.3. Containment material

The design of a thermal storage system implies a careful selection of materials which will be involved, not only PCM itself but also the container. This is a critical step in the design process since the system works under high temperatures and load cycles and a wrong choice can lead to undesirable failures. Chemical compatibility between PCM in its molten state and containment material has to be deeply checked to assure absence of reaction when the system is working. Thermal cycling and mechanical resistance of PCM containment is as well an important issue to consider as well as high temperature creep performance. Regarding the containment material, it should have some characteristics to assure good behavior of final PCM capsules [6,10,15]:

- Meet defined specifications of strength, flexibility, corrosion resistance and thermal stability.
- Act as barrier to protect the PCM from harmful interaction with the environment.
- Provide sufficient surface for heat transfer.
- Provide structural stability and easy handling.
- Control the changes in volume of the storage materials as phase change occurs.

2.4. Encapsulation technology

Apart from the melting temperatures, other parameters such as sizes, shapes, etc. are also to be taken into account. Taking into account thermocline system configurations, initially encapsulations of around 10 mm are considered with a preferred round shape. Anyway, these parameters are being affected by many factors further explained in this report.

High temperature PCM confinement inside a capsule has been studied so far by a few research groups worldwide [15-21], though this concept has been still extensively analyzed for low temperature materials.

The main objective of encapsulation is the enhancement of PCM heat transfer rate due to inherent low thermal conductivity of salt-based materials. On the other hand, while metals and alloys do not have this problem, its encapsulation is also interesting for many reasons such as:

- Holding the liquid and/or solid phase of the PCM and keeping it isolated from the surrounding to prevent reaction or mixing with the HTF.
- Flexibility for volume change of PCM during melting-solidification cycling.
- Enhancement in thermal and mechanical stability of the PCM.
- Improve the compatibility of hazardous PCM that cannot be directly used or immersed.

All of these objectives are necessary for creating the proposed thermal storage system. Besides, encapsulated material can be easily handled and placed where needed inside the storage tank depending on its melting temperature.

Regarding on particle size, encapsulated PCM can be classified as follows: bulk storage in tank heat exchangers, macroencapsulation (>1 mm), microencapsulation (0.1-1000 μm) and nanoencapsulation (0-100 nm) [10,15,22,23].

Bulk storage is referred to the tank heat exchangers for PCM, similar in design to existing tanks used for energy storage. Their main characteristic is the need for a more extensive heat transfer area because of PCM higher heat storage density and lower thermal conductivity compared to other storage media. The most common approaches consist on inserting fins, using high conductivity particles, porous media, metal structures, fibres in the PCM side or direct contact heat exchangers [3].

Macroencapsulation is the most common form of encapsulation currently applied. A significant quantity of PCM (from few grams to kilograms) is encapsulated in a discrete unit. Its advantage resides in its applicability to both liquid and gas as HTF. The shape of the macrocapsules can be tubes, rectangular panels, spheres, or pouches without a defined shape. A successful design of a macrocapsule should fit well with the intended application.

Microencapsulation is defined as a process in which tiny particles or droplets are surrounded by a coating, or embedded in a homogeneous or heterogeneous matrix, to give small capsules with many useful properties. The size of the shells oscillates between 0.5 and 150 μm thickness and the core constitutes between 20 and 95% of the total mass [24,25].

Nanocapsules are structurally more stable compared to macro and microcapsules and there is a great potential in its use for TES applications and also in HTF. This is a recent field of study and research and little scientific production has been done. Development of this new PCM encapsulation option is still at concept-laboratory level. More investigations are required to bring nanocapsules into commercial applications [10,26].

3. Material and process selection

3.1. PCM

The selection of the PCM to be used in a latent storage system as the storage media is directed towards fulfilling a series of desirable properties considering thermodynamic, physical, chemical and economic aspects [3,5,7,22].

When selecting a PCM, its melting point must be related to the plant operating temperature. The volumetric latent heat and the density should be as high as possible to minimize the storage unit size. Minimum change in volume between phase transition and low vapour pressure at operation temperatures are also important. In addition, a high specific heat will provide additional sensible storage capacity. A high thermal conductivity, both in solid and liquid state, is desirable to improve charging and discharging processes and to obtain a more uniform temperature distribution within the storage unit. The PCM must have congruent melting to avoid component segregation and storage properties decrease with thermal cycles. No sub-cooling and enough crystallization velocity are necessary to assure good heat transfer rates.

Chemical stability (oxidation processes, thermal decomposition and material compatibility) under operating conditions is important to assure long lifespan of selected PCM. Great attention deserves also safety considerations such as easy handling and storage, low flammability risks and no toxic materials.

Despite there is a huge amount of PCM available in a wide range of melting temperatures, it is not possible to find a material that fulfils all these features. Therefore, it is necessary to compensate some material properties weaknesses with different solutions such as the use of enhanced heat transfer areas to avoid the problems related to the low conductivity of several PCM materials.

Inorganic salts and metals are the main candidates for heat storage applications at high temperatures (>300 °C) [3,7,22,23,27].

Inorganic salts and their eutectics have been employed as PCM in most of latent storage system proposed by researchers worldwide, because of their low cost and availability. However, these PCM have several disadvantages that limit their application; such as very low thermal conductivity, corrosive nature, non-negligible phase change associated volume increase and considerable subcooling.

On the other side, metals and alloys appear as feasible candidates. They have been not seriously considered as PCM yet though they represent a promising option compared to inorganic salts. They possess high thermal conductivity, high latent heat of fusion per volume unit (reducing volume of storage system) and low vapour pressure. Their main disadvantages are their higher cost and density in comparison to inorganic salts.

A deep search has been conducted across high temperature PCM literature [4,5,22-31] to find the best materials regarding melting temperatures, latent heat of fusion, thermal and chemical stability, safety and environmental issues. Materials fulfilling these features are both inorganic salts and metals. Final candidates and their properties can be found in Table 1.

Table 1. Selected PCM and their properties, containment materials and encapsulation methods.

Melting temp,	PCM (% wt)	T _m , °C	ΔH _f J/g	Containment	Encap. method
295 – 315 °C	NaNO ₃	308	177	C. steel, S. steel	External coating
370 – 390 °C	KOH	380	150	Ni 200 / 201	External coating
	Zn/Al/Mg (95.96/4.00/0.04)	381-387	110	Haynes 188 and 556	Preformed shell
	Zn/Al (95.96/4.00)	381	138	Haynes 188 and 556	Preformed shell
540 – 560 °C	(Li,Na,K) ₂ CO ₃ (20/60/20)	550	283	316 S. steel	External coating
	Al/Si/Mg (83.14/11.70/5.16)	555	485	Ti and its alloys Ceramics	Preformed shell
	Al/Cu/Mg (70.60/25.46/3.94)	560	545	Ti and its alloys Ceramics	Preformed shell

3.2. Containment material

Regarding corrosion aspects, structural material should withstand molten salts or liquid metals, which represent a concrete case of corrosive liquid environments. Much attention has been paid on them from nuclear industry, where the major research has been made though it is an important issue affecting other different industries.

Corrosion damage by molten salts can occur in a wide variety of materials and by different mechanisms [32-34]. Fused salts can cause corrosion by the solution of constituents of the container material, selective attack, pitting, through electrochemical reactions, by mass transport due to thermal gradients, by reaction of constituents of the molten salt with the container material, by reaction of impurities in the molten salt with the container material, and by reaction of impurities in the molten salt with the alloy. Although many hundreds of molten salt-metal corrosion studies have been documented, quantitative data for materials selection and performance prediction are rarely available [32]

There is a lack of reliable data for molten salts on structural material under industrial conditions, but several general rules should be observed. A material should be selected that will form a passive non-soluble film in the melt if possible. Material selection is the key to successful containment. Minimizing the entry of oxidizing species such as oxygen and water into the melt is very important. The oxidizing power of the melt or its redox potential should be kept as low as possible, which can be accomplished by additives. Temperature gradients should be eliminated within the melts to decrease the selective dissolution and plating at hot and cold sites.

Liquid-metal corrosion [32,33] differs fundamentally from molten-salt corrosion in that the medium, except for impurities, is in a nonionized state. The solubilities of the alloy components and their variation with temperature then play a dominant role in the process, and preferential dissolution is a major form of degradation. Mass transfer is

another frequent consequence of the dissolution process. At the same time, the corrosion is strongly affected by the presence of non-metallic impurities in both the alloys and the liquid metals.

Scientific papers of corrosion in molten salts and liquid metals have been thoughtfully reviewed in order to propose potential containment materials. Initially, general behavior rules for similar compounds (e.g. nitrates, carbonates, aluminium alloys) were analyzed, obtaining a close idea of possible materials to be employed with. Besides, specific corrosion studies with selected PCM have been found in most cases, assuring theoretically their feasibility. Nevertheless, any data or information appearing here should have to be experimentally proved at a lab-scale before designing any thermal storage system. Table 1 shows final containment materials proposed for each case.

3.3. Encapsulation technology

In general terms, the process of enclosing a PCM inside a shell can be developed considering two main approaches:

- Inserting the PCM into a preformed shell.
- Developing an external coating over a PCM pellet.

The first concept is quite simple; it basically consists of filling an empty capsule with the desired PCM and then sealing it hermetically to avoid PCM leakage. Good shell and core material compatibility and free space to allow PCM volume expansion should be guaranteed. Shell thickness and geometry can influence final thermal behaviour of PCM-shell joint.

A second alternative is the deposition of an external layer around a PCM pellet using traditional coating techniques. A PCM pellet of desired material, shape and porosity is fabricated and a coating or a series of coatings are deposited onto the pellet which would act as a shell. The PCM optionally includes a void space within the pellet permitting the phase change material additional space during melting. The porosity of the pellet depends on the shape and the operating conditions applied during the pelletizing process [35,36].

It is known that a metal shell or a coating around PCM implies increasing effective thermal conductivity of the storage medium, higher thermozone regions and lower efficiency as demonstrated by Bayón et al. [37] but this issue will be evaluated in a further step both experimentally and with simulations.

3.3.1. Inorganic salt PCM

The main disadvantage of most salt-based PCM is its low thermal conductivity. For this reason, it is interesting to confine salt in a volume as small as possible and the best option consists of developing a shell around a preformed PCM pellet. As stated above, salt based PCM require small sized encapsulations to have a good heat transfer between the external melt salt and the internal PCM to guaranty a high percent of phase changing. The most appropriate methodology to reach this goal is the creation of an external coating According to the models and simulations previously developed by project partners [1,2], the proposed thermal storage system would be fully optimized with spherical-shaped encapsulated PCM with a diameter of 10 mm.

Due to hydrophilic nature of inorganic salts, it is not possible to make a direct coating of metal, metal oxide or ceramic over the capsule. Consequently, an intermediate polymeric layer is essential in order to avoid solid salt dissolution. This polymer or inorganic material can be applied in a thin layer onto the salt pellet by dip-coating, spray coating or brushing [26].

Polymers with the best characteristics for encapsulating the PCM are the following:

- Degradable flexible plastics: for example, PMMA or PEMA. They act as a sacrificial layer which is removed by heat treatment and leads to a void space between the PCM and the shell that will allow the expansion of the PCM when melting and protect the PCM from dissolving in water as well as in several organic solvents. This route has been also found in the state of the art analysis by University of South Florida [13,14].

- High temperature polymers: for example PI. They make the salt PCM more stable when it melts at high temperatures.
- Conductive polymers: for example, the use of PANI (polyaniline). They make the salt PCM more stable when it melts at high temperatures and more conductive so that the further process of electroless or electrochemical deposition becomes easier.

The exterior of the polymer-coated pellet can be then encapsulated with a metal coating by electrolytic or electroless deposition processes. In that case, the use of conductive polymers or the presence of a conductive element in the polymer layer is required, which can be incorporated by dip-coating together with the polymer as described in [26,27]. In general, most of the metals (such as Ni, Cu, Co, Cr...) are electrodeposited from aqueous solutions.

In a different approach, the encapsulation of the salt-based PCM in a metal oxide shell can be carried out by the sol-gel method [26,27]. Metal oxide encapsulants include SiO₂, TiO₂, ZnO, sodium silicate and others. The most common sol-gel technique is carried out in aqueous solutions so that the intermediate coating is necessary, but not the presence of a conductive element.

In principle the building up of final metallic shells rather than final metal oxide shells by the sol-gel method seems more interesting since the final metal shells would guarantee the strength, heat conductivity and ductility of the final material. The main issue in this case would be associated to corrosion respectively between the metallic shell and the PCM and between the metallic shell and the external environment (melt salt of thermocline tank).

The most straightforward solution seems to start with a PCM pellet material with adequate porosity (permitting the PCM material additional space during melting), then applying a conductive polymer coating using a nonaqueous solution/dispersion in order not to dissolve the PCM and then follow with the application of the desired metal coating by electroless or electrolytic deposition process. Anyway, the suitability of this encapsulating method highly depends on the specific salt based PCM and the required encapsulation materials.

An initial estimation of the encapsulation costs has been done considering a 50 µm thickness nickel coating over 10 mm diameter spheres. Polymer coating has not been included in this evaluation yet. As an example, this means a cost of 60 €/kWh for NaNO₃, considering also the salt cost. After doing these cost estimations, a further and deeper analysis is found to be required. It needs to be understood that this would be the cost for a laboratory scale process. For an industrial, large scale process, this cost will be greatly reduced. Industrial application of Zn encapsulations may be easier than Ni but, this needs to be validated by corrosion tests yet, so estimations in Ni are reported here. The calculated estimations are higher than the values published by Terrafore [38] (5 \$/kWh) and thus, a further and deeper analysis is required. This needs to be addressed in detail. Achievable costs at industrial scale processes need to be assessed to see if the creation of external coatings by the most interesting methods is economically viable.

3.3.2. Metallic PCM

Due to this high thermal conductivity, a metallic PCM may be encapsulated in bigger structures guarantying a good phase change performance. Therefore, the first approach comprising a preformed shell can be followed. Different capsule geometries can be used in this methodology; spheres, cylinders, cones, rectangular or triangular prisms or specially designed geometries. From one side, high values of surface area to volume ratio are required to maximize the heat transfer from the external melt salt to the internal metallic PCM. In this sense, spheres are penalized. On the other side, higher surfaces mean higher encapsulation material needs. In this research the cylinder geometry is selected due to manufacturing, sealing and material availability aspects.

There are different possible methodologies to introduce the PCM into the encapsulation cylinders. It could be an option to fabricate bars of a certain dimension of the selected PCM and then introduce them into the preformed cylinders. Anyway, it is considered that the most affordable and common technique is pouring-casting the melt PCM into the encapsulation shells and then sealing them by a common welding process.

Regarding the mechanisms to handle with the internal pressures of the encapsulations, mainly two different options are seen.

- Flexible encapsulation material completely filled with PCM; elastic deformation of the container material; deformation of the encapsulation depends on the volume variation of the PCM during phase change.

- Stiff encapsulation materials. A gas volume must be included in the container to compensate volume variations of the PCM.

The employ of flexible encapsulations are much more complicated. A possibility could be designing encapsulations with minimal wall thicknesses leading to high elastic deformations. Another feasible solution would be using elastic encapsulation materials but no material has been found for such high temperatures. In this sense, stiff encapsulations are selected with a certain gas volume for the expansion of the PCM when melting. The thickness of the encapsulations needs to be optimized taking into account different aspects such as: corrosion rates, cost of material, weight, etc. A brief economic evaluation of this option has been performed, considering the price of heat storage alloys and metallic tubes chemically compatible with them. Manufacturing process of inserting the alloy in its liquid state, capping and welding is also included. Global costs are around 700 €/kWh for Zn-Al alloy and 400 €/kWh for Al-Si alloy. Industrial suppliers and manufacturers of metallic tubes have been contacted to calculate this values.

The specific encapsulation method selected for storage materials in the system can be found in Table 1.

4. Conclusions

A theoretical design of PCM encapsulations of a thermocline thermal storage system for CSP has been developed. According to the system requirements, several PCM have been selected along with inert materials to contain them. Encapsulation procedures have been also technically evaluated for every option. It has been appreciated that encapsulation of these high temperature PCM is a complex field and a deep analysis of possible alternatives have been performed. Designing work is currently going ahead by developing an accurate estimation of encapsulation cost at industrial level for all the selected PCM and structural materials. This is a critical issue to define the best material and to evaluate the economic interest of the proposed system. Having this into account, it is envisaged that the best encapsulation techniques are using preformed shells for metallic PCM and creating external coatings for the salt PCM.

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