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REVIEW ARTICLE

Materials and Selection Thereof for Encapsulated Phase Change Materials for Heat Transfer Applications

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HIGHLIGHTS

- > Material selection for encapsulated phase change materials has emerged as a standalone field of study.
- > The current state of technology in micro/nano encapsulation of PCM enable researchers conduct multidisciplinary studies in a variety of fields of industry, including but not limited to advanced heat transfer fluids and thermal energy storage.
- > Specialization in the development of new and more advanced PCM core and shell materials and in selecting the optimum material for any demanded need is believed be an important career building option.

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ABSTRACT

The rapid expansion of Phase Change Materials in different application areas and the increase in the diversity of the commercially available Phase Change Materials have made selecting the most appropriate and efficient material in a given application a field of study. In recent years, new heat transfer fluids are prepared by using phase change materials to form solutions with various carrier fluids by encapsulating them in micro or nano dimensions. There are great research and career opportunities in the development of novel encapsulated PCM and PCM slurries with new, advanced PCM materials. As well as developing new advanced encapsulated PCM, the optimal selection thereof is crucial and is believed to be a separate field of specialization. This study is aimed to present a brief summary of phase change materials, and their advantages and drawbacks, as well as the principles and methods in the selection thereof.

Contents

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1. Introduction

In recent years, Phase change materials [PCM], which has become focus of interest in many fields, especially in the field of thermal energy storage, heating-cooling, ventilation and air conditioning, and temperature regulation, has seen a great progress under the light shed by studies conducted on the chemical properties of these materials, their performances in various applications, and on the synthesis and characterization in order to meet different needs. The development of encapsulation techniques has enabled highly efficient and versatile studies.

Phase Change Materials, as their name denotes, are organic or inorganic compounds that undergo a phase change in a given temperature range. A phase-changing substance absorbs or releases, as much thermal energy as its latent heat of fusion, from the environment at constant temperature. Therefore, it is very important to obtain the PCM having a phase change temperature and latent heat of fusion of which required by the application. Figure 3 shows Phase Change Materials in category-wise with their melting enthalpy versus melting temperatures.

2. Phase Change Materials and General Properties

In PVT systems, electric and thermal energy are obtained simultaneously, but the conversion of these two energies into useful work may not take place simultaneously, which is the case particularly in thermal energy recovery, the resulting energy must be stored.

Thermal energy can be stored as thermochemical heat, sensible heat or latent heat [1]. Thermochemical heat storage is based on the occurrence of reversible endothermic / exothermic chemical reactions, which in most cases are not feasible. In sensible heat storage, since the materials used reversibly exchange heat, they are required to have a high specific heat capacity, have long service life, and to not be corrosive or toxic[1, 2], as well as required to have been isolated and insulated during storage. And latent heat storage is defined as the storage of energy in the material at a constant phase transition temperature.

Due to the fact that PCM have the ability to exchange heat at constant temperature and store the energy they receive in the form of latent heat [3, 4], they offer higher energy storage density [5] as well as provide considerable advantages particularly in systems where an optimum operating temperature needs to be maintained [6].

The idea of using PCM, which draws heat at constant temperature in the form of latent heat so as to achieve a stable operating regime and to ensure the storability of the drawn heat, is not very new, but it is not much studied either.

In such a heat transfer process seen in Figure 1; in the case of a phase change that takes place at a temperature of (T_p) between the initial temperature (T_i) and final temperature (T_f), the total amount of heat transferred is greater than that of the same process without a phase change by the latent heat of fusion (H_f), and is obtained by Equation 1 [7].

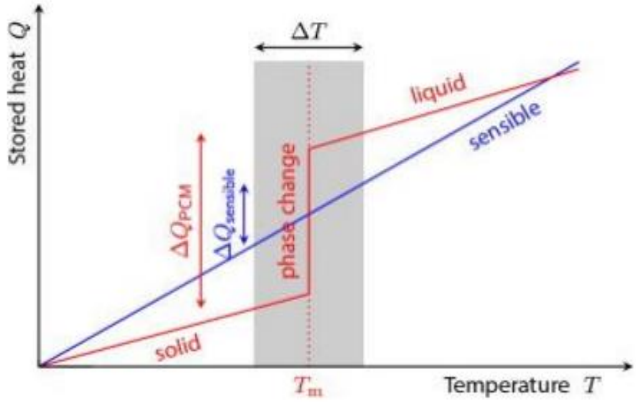


Figure 1 The amount of stored heat at a given ΔT temperature range in sensible and latent heat storage cases [8, 9]

$$\int_{Tp}^{Ti} mC_p dT + mH_f + \int_{Tf}^{Tp} mC_p dT = m\,[H_f + C_s(T_p - T_i) + C_l(T_f - T_p) \qquad (1)$$

where C_l is the specific heat capacity of the liquid phase, C_s is the specific heat capacity of the solid phase, and C_p is the specific heat capacity at constant pressure.

When the ambient temperature reaches the phase change temperature of the PCM, the PCM starts changing phase during which it continuously absorbs or releases heat from or to its environment depending on the direction of phase change. The heat absorbed or released by the PCM during the phase change is associated with the chemical structure that holds the material in its physical state in that phase, and hence no change occurs in the sensible temperature, hence the temperature of the material, during the phase change. In the absence of a significant change in ambient temperature, PCM materials offer effective solution alternatives for heat storage and temperature stabilization thanks to their capabilities of absorbing and releasing a large amount of latent heat [1, 3].

PCM materials with fusion energies varying in a wide range of -5 °C to 150 °C are available [10]. On the other hand, compared to sensible heat storage systems, PCM increase the amount of energy that can be stored per unit volume by 5 to 14 times [11].

PCM, which we want to take advantage of the latent heat storage capacity, have to provide some thermodynamic, kinetic, physical and chemical properties so as to serve for the purpose.

3. Classification of Phase Change Materials

Generally, the phase change materials are classified into four types as solid-liquid, solid-gas, solid-solid and liquid-gas, as shown in Figure 2. Liquid-gas phase change materials have a higher latent phase change heat than other phase change materials, but solid-liquid phase change materials are mostly preferred because of the problems experienced in storage of gaseous PCM due to their large volumes [11].

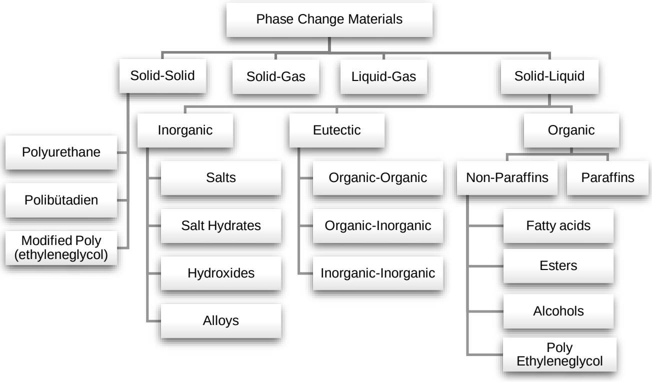


Figure 2 Classification of Phase Change Materials [5, 6, 12]

Hundreds of studies conducted to date have investigated the usability of thousands of substances as PCM in single or mixtures, and the various properties of these materials such as chemical, thermal and physical properties have been determined and published in the literature. The chart in Figure 3 that shows melting temperatures and enthalpies per material class was obtained from the results of such studies.

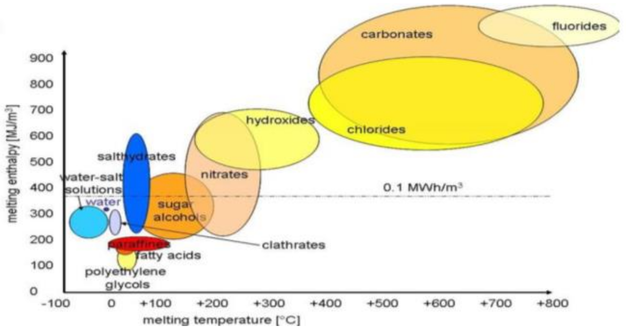


Figure 3 Phase Change Materials according to Melting Temperatures and Enthalpy [4, 6, 13]

Solid-liquid phase change materials are divided into three sub-classes as Organic, Inorganic, and Eutectic.

3.1. Organic Phase Change Materials

3.1.1. Paraffins

Pure paraffins consist only of alkanes and may be straight-chain (n-alkane) or branched (iso-alkane) depending on the chain length of the alkane. The number of carbon in the structure of the paraffins, which are in the general chemical formula of C_nH_{2n+2} and which consist mainly of heavy hydrocarbons, is the determinant of the phase in which paraffin is normally present. Up to five carbons are present in liquid form between five and fifteen carbons and in solid form in the form of waxes with higher carbon. Melting temperatures and melting latent temperatures of alkanes increase with increasing carbon number. Melting points of C_{14}-C_{40} alkanes are in the range of 6-80 °C [2].

Paraffins are the most widely used PCM material. It is frequently preferred due to its relatively low thermal conductivity compared to other alternatives and its more attractive features despite its high cost of approximately 1,400 USD/ton. In particular, its superior thermal properties, such as stability in thermal cycles, make paraffins the most applicable PCM in systems such as PV and PVT that heat and cool daily between significant temperature differences. [3, 14, 15].

Table 1 Advantages and Disadvantages of Paraffin [2, 6]

Benefits	Drawbacks
No phase separation	Low thermal conductivity
Chemical stability	Combustible
It does not tend to sub-cooling, so no nucleating agent is required.	Does not have a constant melting temperature
High melting latent heat	
Non-corrosive and non-toxic	
Low cost	
Easy to supply	
Low vapor pressure	
No large volume changes during melting	
Melting points are within a wide range.	
Low vapor pressure	
No phase separation	

Although they possess a number of sought properties, such as negligible sub-cooling, chemical stability, high latent heat, low vapor pressure, no phase decomposition, spontaneous nucleation and commercial presence; major volumetric change during phase change, low thermal conductivity, and flammability are the main disadvantages of paraffins, and organic PCM in general [6].

3.1.2. Non-paraffin

A wide variety of organic phase change materials such as esters, fatty acids, glycols and alcohols fall within this group. They exhibit excellent melting/crystallization characteristics without sub-cooling.

Among the non-paraffin organic phase exchange materials, fatty acids in particular, have very similar characteristics to those of paraffins. The fatty acids, which takes this name by virtue of the fact that they are obtained from edible vegetable and animal origin triglyceride oils, as it is the case in the paraffins too, have increasing viscosity with increasing carbon numbers, hence they are present in the solid state in high carbon numbers. Low-carbon fatty acids that exhibit poor acidity are water-soluble and have high surface tension in the liquid phase, which is capable of being retained in the structure of an adsorbent material such as polymer or clay. In addition, fatty acids do not possess any negative properties such as being corrosive, tendency to sub-cooling and instability, and they possess all the thermodynamic, kinetic, chemical and thermal criteria sought in energy storage materials [2].

3.1.3. Inorganic Phase Change Materials

The major species of inorganic PCMs are salt hydrates. Although they have negative properties such as being corrosive, tendency to sub-cooling and instability, they are attractive due to their properties, such as having high latent heat of fusion and high water content, being cheap and non-flammable. While there are studies focused on eliminating their negative properties, still organic PCMs are preferred as alternatives in general.

3.1.4. Eutectic Phase Change Materials

Eutectics are phase change materials consisting of two or more components, that is they are mixtures composed of two or more chemical substances.

Based on the constituent types, they are classified as organic-organic, inorganic-inorganic, and organic-inorganic mixtures. The idea behind the eutectics is to attain the ability of acquiring the demanded melting temperature that is required by a specific application. The composition of eutectics are so flexible that a range of 25–832°C melting temperature can be achieved, and a similar latent heat of fusion as that of organic PCM is obtained [12]. However, experimental studies to determine thermo-physical properties are not yet sufficient.

Eutectic mixtures of inorganic salt and water form compounds that behave like water but solidify and melt at lower temperatures than water, and whose thermal conductivity is similar to water. At this point of solidification, known as eutectic temperature, sub-cooling usually occurs, but this phenomenon can be prevented or reduced by a variety of applications which are also mentioned in this article. Also, the eutectic water-salt compounds, just as water, exhibit a significant volume change of 5% to 10%, Another disadvantage of these chemically stable compounds is that they are corrosive to certain materials such as metals [6].

Table 2 Main advantages and disadvantages of solid-liquid phase change materials

Class	Benefits	Drawbacks
Organic	Available in a wide temperature range	Low thermal conductivity (approx. 0.2 W/mK)
	High fusion temperature	Relatively large volumetric change
	Low overcooling	Flammability
	Chemically stable and recyclable	
	Good compatibility with other materials	
Inorganic	High fusion temperature	Sub-cooling
	High thermal conductivity (approx. 0.5 W/mK)	Corrosion
	Low volume change	
	Low volume change	
	Low cost availability	
Eutectic	Sharp melting temperature	
	High volumetric thermal storage density	Lack of existing test data for thermo-physical properties

4. Microencapsulation and Encapsulation of Phase Change Materials

PCM materials are not always suitable for use in circulation circuits. Especially at the point of benefiting from solid-liquid phase changes, some drawbacks/problems, such as leakage [3] or precipitation, are experienced, which restricts their use in more advanced applications. One way to solve the problem is coating PCMs with different shell materials [16], and obtain kind of a composite material. Therefore, PCM are widely used in capsules (generally of micro or nano scale) to prevent leakage and to maintain the effectiveness of thermo-physical properties when applied in slurry active systems [17], despite the fact that encapsulation adversely affects system performance due to the increased thermal resistance [6].

Encapsulation is a process in which particles of solid or liquid material are coated with polymeric materials. The project to produce carbonless copy paper by the National Cash Register Company (NCR) is considered to be the first study on encapsulation [18].

The shell of the capsules prevents the core material from interacting with the carrier liquid, which increases the stability of the material and prevents aggregation of the particles. For this purpose, encapsulation technology has been widely applied in pharmaceutical, chemical industry and biological engineering for many years. The development of technology over time has led to the search for solutions for new problems and the use of capsules in a growing number of fields, and today, a wide range of capsules can be produced in nanometers to millimeters.

Capsules by size are divided into three groups [19], and classified roughly as:

- Nanocapsules (sizes between 1 and 1000 nm)
- Microcapsules (sizes between 1 and 1000 µm)
- Macro capsules (sizes greater than 1mm)

Encapsulation processes are also called macrocapsulation (or encapsulation), microencapsulation, and nanoencapsulation depending on the capsule size to be obtained as a result of the process.

Micro/nano-encapsulation is a process in which very small particles or droplets of core material are wrapped or coated with a film, and embedded into a homogeneous or heterogeneous matrix [12, 20]. The particles in the core may be liquid, solid, gas or multiphase, and the film acting as a shell is generally a polymer [21]. The materials thus produced are called encapsulated PCM (ePCM). Encapsulation improves thermal and mechanical properties of PCMs [22], increase heat transfer surface area [16], hence the surface to volume ratio, and significantly improves the thermal capacity and heat transfer efficiency of heat transfer fluid [1]. In addition, the capsules allow the materials to be used as solid particles in the liquid state and compensate the volume change within the shell during the phase change.

In recent years, many studies focusing on the preparation and properties of suspensions, which are prepared by mixing capsules having PCM as its core material with some carrier fluids, have been launched and important results have already been obtained [23].

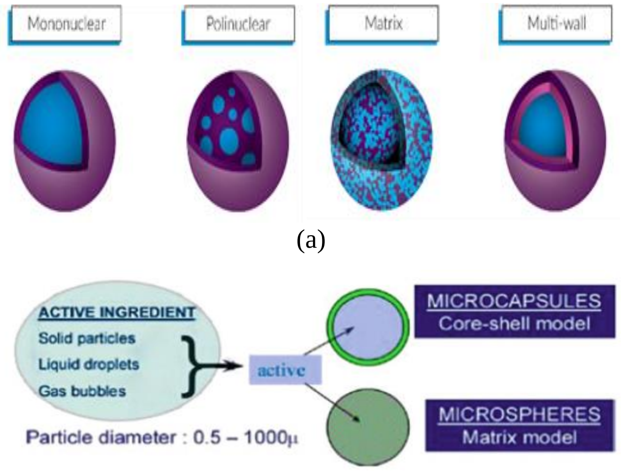
It is also known that the problems of PCMs include sub-cooling, and low nucleation and crystal growth rate. The size of liquid droplets after nucleation is a major factor affecting crystal growth. Encapsulation is also used as a method of addressing these problems. Inasmuch as the ability of producing smaller diameter capsules is made possible with the developing technological means, these problems of PCMs can eliminated to that extent. Studies have shown that the size of the microcapsules has a direct effect on the crystallization temperature and that the overcooling temperature is inversely proportional to the capsule diameter for capsules with a size of 5-100 μm [24]. In addition, Cao and Yang [25] have developed a new technique to suppress sub-cooling in microencapsulated PCMs (mePCMs) by optimizing the composition of the microcapsule resin shell .

Figure 4 Phase change of PCM material in capsule during heat exchange [26].

In Figure 4, the composite structure in the form of a shell and core after encapsulation and the phase change of PCM within the shell during heat exchange are given.

In this way, problems such as precipitation and decomposition which may occur in the mixture can be solved by taking advantage of the different properties of the shell material and the leakage of the core and mixing with the carrier fluid are prevented.

It is possible to obtain encapsulated materials of different morphological structure using different encapsulation methods, combinations of core and shell materials. This is also dependent on the physical–chemical properties of the core, the wall composition [20]. In Figure 5, (a) illustrates different types of capsules with single-core, multi-core, matrix and multi-layer shell structures, and (b) illustrates the typical structure of an encapsulated phase change material having the form of either a microcapsule or a microsphere.

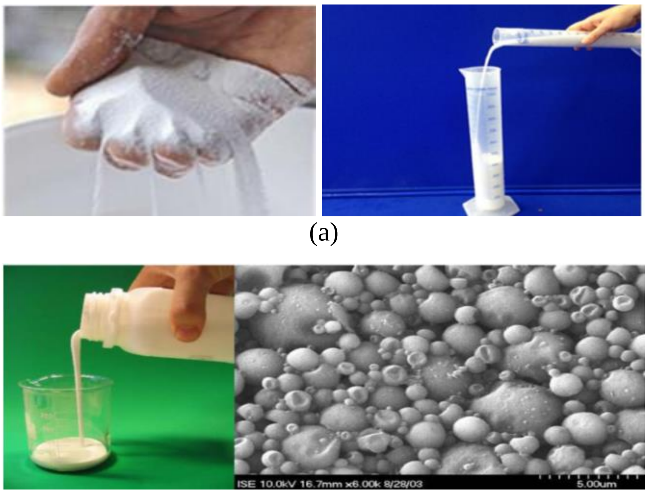


(a)

(b)

Figure 5 (a) Morphology of encapsulated materials [27] (b) Typical structure of an ePCM [20]

The capsule structure to be preferred in heat transfer applications depends on the amount of heat to be drawn from the system per shell and core material, the stable operating temperature of the system and a number of other system parameters. However, one of the most important points is that the phase change temperature of the core material to be selected is close to or below, but not too far, the stable operating temperature of the system, and that the shell material is not damaged, broken or torn during pumping. Figure 6.(b) shows instances of broken capsules during pumping. A capsule size between 2 to 10 μm was reported to ensure that almost no microcapsules are broken in durability tests [28, 29]. On the other hand, centrifugal pumps are reported to have been able to pump the slurries in longer periods without deforming the microcapsules than do other pump types [30].



(a)

(b)

Figure 6 (a) mePCM in powder form and in slurry form [31] (b) mePCM its slurry and capsules many of which are broken due to pumping [13].

Smaller capsule sizes will result in greater surface area, better suspension stability and lower fracture rate. Another problem, precipitation and decomposition, can be solved by using different surfactant mixtures.

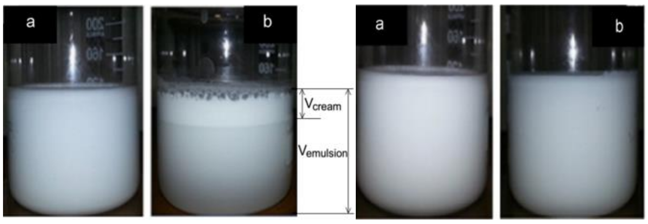


Figure 7) Decomposition in the PCM emulsion with single surfactant (left) and different surfactant mixtures (right), at the initial state (a) and after 1 day (b) [27].

4.1. Selection of Shell and Core Materials

Since there should be no chemical interaction between the shell material and the core materials, therefore chemically compatible [12], the selection of the shell materials and core materials should be selected together depending on the intended use and each other. The characteristics of the material to be encapsulated are very different and varied, and the selection of a pair of core-shell materials that can withstand the physical conditions and operating conditions of both the core material and the environment in which the capsule will be used is a very laborious and meticulous process.

The shell material is effective on the mechanical strength of the ePCM as well as on the heat transfer properties. Shell material with good thermal properties and high strength will not only improve the thermal performance of the system, but will also increase the thermal cycle life of the ePCM [19].

On the other hand, although there are various components / materials that can be used as a shell material, in a typical encapsulation process, formaldehyde resins, such as urea-formaldehyde resin, are generally used. However, some of the byproducts, residues or wastes resulting from the encapsulation process contain components that are harmful to the environment and human [32]. This takes place in the processes and applications as a constraint parameter that should be avoided in terms of both green technologies and environmental sensitivities.

Materials to be selected as shell material:

- should have a good degree of flexibility,
- must be sealed to prevent ITA penetration and ensure that the core does not leak,
- must be stable enough to maintain structural and thermal stability in phase change processes,
- must have low reactivity to both the carrier fluid and the core material,
- must retain the thermophysical properties it bears in macroscale in micro- and nano-scales, too,
- should have high thermal conductivity so as not to hinder/delay heat transfer between ITA and PCM [19].

Many organic and inorganic materials having these properties are suitable for use as shell materials, but the most commonly used shell materials are polymers. Polystyrene, polymethylmethacrylate, gum arabic, gelatin, amino plastics, arabic gelatin-gum, urea formaldehyde resin, melamine formaldehyde resin, gelatin formaldehyde resin and polypropylene, polyolefin, polyamide and the like are selected as shall material [19, 33].

The selection of the core material to be used in the production of encapsulated phase change materials is also very important and is a process that must be carried out meticulously by considering a large number of parameters. As with shell materials, core materials too must possess some key features, apart from those that apply in accordance with the intended use and environmental and operating conditions. These can be listed as: (1) appropriate phase change temperature or temperature range, (2) high phase change latent heat, (3) good thermal conductivity, (4) low density/volume change between phases and (5) low reactivity [1, 2].

Table 3 Main characteristics that a typical core PCM is required to have [16]

Category	Property
Thermal	Melting point in the desired operating temperature range
	High latent heat capacity value
	High thermal conductivity
	High specific heat value
Kinetic	No super-cooling
	No sub-cooling
	No phase segregation
	Good nucleating properties
Chemical	Chemically stable over a no. of freeze thaw cycles
	Non corrosive
	Non toxicity; non flammability
	Compatible with container material
Physical	Small vapor pressure
	Small phase transition volume change
	High density
Others	Low cost
	Easily Available

When the studies in the literature are examined, it is seen that paraffins, in the solid-liquid phase organic materials group, are the preferred and practical material for the production of encapsulated phase change materials. Although inorganic phase change materials provide advantages such as high thermal conductivity and high energy storage density, it can be said that they are not preferred because of the sub-cooling and phase separation during the phase change from liquid to solid. Ideal core materials can be obtained from mixtures of organic or inorganic PCM materials to take advantage of both groups of materials [23], which were previously mentioned as eutectics.

Optimal selection of the most suitable PCM shell and core materials for the application required is a time-consuming and exhausting process that needs to be scrutinized. As the application areas of PCM and the need for microencapsulated PCM increase, easier and more precise conduct of this process has emerged as a standalone engineering problem per se. Barreneche et al. [34], using CES Selector, an Engineering Materials Selection Program developed by GrantaDesign, which was established in 1994 in Cambridge University Faculty of Engineering and joined Ansys in early 2019, has prepared a database that can be used in the optimal selection of PCM according to their Chemical structures, properties and application areas. Thus, the selection of PCM can be facilitated for any application.

4.2. Commercially Available Encapsulated PCM

The rapid increase in the demand for "encapsulated phase change material research" has brought upon the opportunity to the industry leaders to build market for ePCM. Today, commercially available ePCMs, manufactured by numerous companies across the world, have been used frequently for different application in low temperature and medium temperature ranges [6].

Several studies have already listed numerous commercially available encapsulated PCM [6, 35]. PCM37 of Microtek Laboratories (paraffin (n-eicosane)), MikroCapsPCM70 of MikroCaps (paraffin wax) [13] Micronal DS of BASF [36], ClimSel C of Climator AB, AC of Cristopia Energy Systems, AC, AN and SN of Environmental Process Systems Ltd are only some.

5. Conclusions

Since appropriate and complete thermo-physical data are not available for PCMs, the relative performances for application-specific purposes of these PCMs is not available in the literature either. In addition, making a good selection of PCM appropriate for any given application among all available alternatives that number in thousands, will only be possible by having theoretically or empirically validating the performance thereof, which is quite unlikely. The selection of the most suitable material is always an engineering problem and is usually done based on the availability of experimental data, expert judgment, and experience. But, most of the times, if not always, these choices are often far from optimal.

In applications where material selection is crucial and reaching optimum is an important indicator, selection procedures should be performed by using scientific methods and special software and methods. Using the Multi-Criteria Decision Making (MCDM) approach and its sub-branches, Multi-Objective Decision Making (MODM) and Multi-Attribute Decision Making (MADM) techniques, a functional relationship is created between alternatives using Figure of Merit (FOM), and by selecting the alternative with the highest FOM value, application-specific material selections can be made optimally. Engineering Materials Selection Software, developed by GrantaDesign CES Selector, is one that can be used for this purpose.

The selection of core PCM and shell materials solely based on their chemical, physical and thermal properties would be inaccurate in terms of engineering approach. In fact, materials need to be evaluated in many different aspects such as harmfulness to environment or human health, commercial availability, cost, carbon footprint, and energy and water use.

In this regard, we predict that, in parallel with the rapidly growing ePCM research interest, there will be an important opportunity in the industry for the commercial production and sale of micro/nano encapsulated PCMs. We also believe that specialization in the development of new and more advanced PCM core and shell materials that are harmless to environment and human health and in selecting the optimum material for any demanded need will be an important career building option.

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