Review on the methodology used in thermal stability characterization of phase change materials

Gerard Ferrer[1], Aran Solé[1], Camila Barreneche[1,2], Ingrid Martorell[1,3], Luisa F. Cabeza[1]

[1]GREA Innovació Concurrent, Edifici CREA, Universitat de Lleida, Pere de Cabrera s/n, 25001, Lleida, Spain, Phone: +34-973-003576, Fax: 34-973-003575, e-mail: lcabeza@diei.udl.cat

[2] Department of Materials Science & Metallurgical Engineering, Universitat de Barcelona, MartíiFranqués 1-11, 08028, Barcelona, Spain. Tel: +34-934021298. e-mail: cbarreneche@ub.edu

[3]Serra-Hunter fellow

Abstract

In general, PCM are classified in organic and inorganic groups or families. First group mainly encloses paraffin, fatty acids, and sugar alcohols. Inorganic are mostly represented by salt hydrates, salt solutions, and metals. Eutectics and mixtures are also being formulated to obtain a desired phase change temperature. One of the most important PCM requirements is being stable after a number of repeated melting/freezing cycles, which is known as cycling stability. A PCM should present the same or almost the same thermal, chemical and physical properties after a repeated number of freezing and melting cycles. Thermal cycling tests results and detailed tests procedures are classified by PCM type in this review. Moreover, the parameters that must be considered in order to perform cycling stability tests are highlighted depending on the importance they have on the following four issues: the choice of the equipment to perform the cycling tests; the selection of the techniques to characterize the PCM before and after thermal cycling test and to follow the PCM thermal degradation; the definition of the number of cycles to perform; and finally, the choice of the heating rate and thermal cycling method (pyramid, or dynamic, or others) to perform the tests. It is mandatory to conclude that, based on the literature reviewed, no common standard for thermal cycling stability tests is available at the moment.

Keywords: phase change materials (PCM), cycling stability, thermal stability, differential scanning calorimetry (DSC), thermal cycler, thermal energy storage (TES)

1. Introduction

Current trends in energy supply and use are patently unsustainable – economically, environmentally and socially [1]. Energy policies are focusing on achieving net zero buildings and on reusing waste heat from the industry. Then, fossil fuel and electricity consumption can be decreased and consequently, CO_2 emissions too. Furthermore, the use of renewable energies is pushing to design new technologies implementing them as a primary source. One huge field implementing waste heat utilization and solar energy is thermal energy storage (TES).

One way of storing heat is by using the latent heat of phase change of a substance, usually from solid to liquid, as it can provide high energy densities. Then, when this stored heat is needed, it can be released by leaving the material temperature decrease, becoming a solid again. Materials used for this purpose are known as phase change materials (PCM). PCM are being implemented in different systems, active or passive, and for several applications, cold storage, building comfort, medium and high temperatures [1-5].

In general, PCM are classified in organic and inorganic. First group mainly encloses paraffin, fatty acids, and sugar alcohols. Inorganic are mostly represented by salt hydrates, salt solutions, and metals [3,6]. Eutectics and mixtures are also being formulated to obtain a desired phase change temperature. All these materials present advantages and disadvantages, as exposed in Table 1.

Moreover, to overcome some of these disadvantages, composites, shape-stabilized or form-stable PCM are being formulated and developed. Composites are mainly thought to enhance thermal conductivity, increase cycling stability, and prevent leakage and corrosion [7-10]. In addition, the combination of nucleating agents with PCM is presented as a solution to decrease subcooling.

The main requirements from the material point of view are, of course, high phase change enthalpy and a suitable phase change temperature, depending on the application in which the PCM needs to be implemented. Furthermore, a PCM is suitable for applications if it is stable after a number of repeated melting/freezing cycles, that is to say, if it has a proper cycling stability. Sometimes it is also called long term stability [5]. This stability encloses thermal, chemical and physical properties, which should remain constant or almost constant after a cycling stability test. Chemical stability is usually studied by infrared spectroscopy (FT-IR) [11]. Another concept is thermal stability. A thermal stability test is useful to ensure that the

PCM is stable at a constant established working temperature. The maximum temperature the PCM can stand is known as the degradation temperature. Thermal stability can be studied in an oven or with TGA [4]. The present review is focused on the thermal cycling stability.

Table 2 reviews the importance given to thermal cycling stability as mentions in different published articles. As shown, and despite the relevance given to these parameters, not much data is available in these publications.

Up to now, few reviews found in the literature highlight the importance of thermal cycling stability, but they do not look at the methodology nor the equipment used in such analysis. Data discrepancy suggests that this property is still not assessed in depth. Moreover, some reviews present only differential scanning calorimeter (DSC) as the analytical equipment, but although DSC is used by all researchers to analyse the materials, it is not always used for cycling them.

Therefore, this paper presents a review on the thermal cycling stability data of PCM found in the literature, but focusing on the methodologies used by the different researchers, along with the equipment used and the analytical conditions in which the tests were carried out in order to find out the existence of a common and useful methodology to develop these tests.

2. Thermal cycling stability testing of PCM

As detailed in the introduction, cycling stability is one key property of PCM to be studied since their lifetime depends on it. A PCM should present the same or almost the same thermal, chemical and physical properties after a repeated number of freezing and melting cycles. Here, thermal cycling tests results and detailed tests procedures are reviewed and shown by PCM type.

It is important to point out that authors just present the results of each consulted study, but do not seek for any phenomena explanation as it is clearly explained in each one of the cited papers. As it is already stated in the introduction, the goal of the present paper is to compile information published in order to find out if there is a common pattern to study the thermal stability of PCM or there is not.

2.1 Paraffin

Table 3 shows the results of the cycling of several paraffin waxes found in the literature. Analytical conditions are first presented, not only the melting properties but also crystallization ones, along with the number of thermal cycles performed. Then, the equipment used for both cycling and properties analysis tests are listed as well as the analysis method used.

Only nine materials are found in this list. The cycled paraffin reported in the literature are both analytical grade and commercial grade, and their melting points range from around 18 ºC to 58 ºC; the numbers of cycles carried out were between 300 and 5000.

All authors show the melting point and the latent heat of fusion before and after the thermal cycling process. Three of the studies, the one performed by Alkan et al. [30], the investigation by Sari et al. [31], both from the same research group at Gaziosmanpaşa University in Turkey, and the research by Silakhori et al. [62], also present information about the latent heat of crystallization. The melting point is taken as the onset temperature on the DSC curve and the latent heat of fusion is calculated using the area under the peak in all cases but the two microencapsulated paraffin studied by Silakhori et al. [62], who do not report the calculating method.

Four different DSC instruments and analysis methods were used in the different investigations to study the thermal properties of the paraffin. Alkan et al. [30] used a Setaram 131 DSC, fixing a heating rate of 5 ºC/min under a constant stream of argon at a flow rate of 60 ml/min (inert atmosphere). Sari et al. [31] used the same operating mode in a Perkin Elmer Diamond DSC. Sharma et al. [26,27] used a Rheometric Scientific Ltd. DSC following a dynamic method. Shukla et al. [29] also used a Rheometric Scientific Ltd. DSC but no specification on the followed operating mode was exposed. However, none of them used the DSC to perform the cycling tests of the samples. Alkan et al. [30] and Sari et al. [31] used a thermal bath setup to melt and solidify the samples by water circulation, while Sharma et al. [26,27] performed the melting-freezing processes using an electric hot plate setup, letting the samples solidify at room temperature. Shukla et al. [29] cycled the samples using an oven and let the paraffin solidify at room temperature as well. Silakhori et al. [62] cycled the PCM in a thermal box equipped with a strip heater and a deep cooler and measured the thermal properties in a Mettler Toledo 820C DSC.

Paraffin wax 53 is the only material presented in Table 3 that has been studied in more than one paper and can lead to some results comparison. The two different studies that Sharma et al.

[26,27] did under the same experimental conditions clearly show that the PCM loses heat storage capacity when cycled. This loss is quantified by the decrease the latent heat of fusion experienced after 1500 cycles, which is around 26%, considerably higher than the 10% suffered after 300 cycles. The melting temperature also showed a 3 ºC decrease after 1500 cycles, a remarkable fact compared to the null variation the PCM's melting point suffered after 300 cycles. Both studies also present the data logged during the cycling process to see the profile the latent heat loss follows and in both cases this loss happens around the mid experimental point, after 100 and 700 cycles, respectively.

2.2 Non-paraffin non-eutectic organic materials

Table 4 presents the results of fifteen non-paraffin non-eutectic organic materials cycled in the literature. These materials include amides, fatty acids, sugar alcohols, and other materials such as urea. Their melting temperatures range from 19 ºC to 216 ºC, and they have been cycled between 50 and 5000 times.

As an overview of the data presented in Table 4, all authors except studies [39] and [36] presented both melting temperature and latent heat of fusion before and after the cycling process. Nikolic et al. [39] only showed the data taken before cycling and oppositely, Abhat and Malatidis [36] only presented the data collected after cycling the samples. Moreover, just four authors out of twelve (33%) also give information about the latent heat of crystallization before and after the cycling process.

Regarding the thermal parameters calculation, the melting point was taken as the onset temperature on the DSC curve and the latent heat of fusion was calculated using the area under the peak in all the studies but the one done by Solé et al [11], where the melting temperature was taken as the peak temperature on the DSC curve, calculating the latent heat with the area under the peak as well. Abhat and Malatidis [36] do not enclose information about the parameters calculations.

Eight different DSC instruments were used in all these different studies but only half of them were also used to cycle the samples. Only acetamide, lauric acid, myristic acid, palmitic acid and stearic acid have been studied by more than one author, thus it is only possible to compare the results regarding these four materials.

Sharma et al. [26,27] studied acetamide as a potential heat storage material. In study [26] the samples were cycled 300 times and in [27] the cycling process was repeated 1500 times. A

Rheometric Scientific Ltd. DSC was used in both studies to find out acetamide thermal properties, following the same DSC method and using a hot plate setup to cycle the samples in both cases. Results show a more important loss on heat storage capacity after 300 cycles (8.4%) than after 1500 cycles (1.2 %), as the obtained latent heat values show. In addition, data in some mid points of the cycle process was also attached to see the evolution that each parameter has with its thermal cycling stability. In both studies, the melting temperature remains quite constant during all the cycling process and the latent heat values do not seem to follow any clear pattern as they increase and decrease randomly with the number of cycles.

Sari [33] (1200 cycles) and Sari and Kaygusuz [40] (910 cycles) studied the thermal properties of lauric acid for its application as PCM. Their studies were done under the same experimental conditions: in both of them a General V4.1C DuPont 2000 DSC was used, following the same 10 ºC/min heating rate in the analysis method and using a thermostatic chamber setup for the cycling process of the samples. The results displayed in Table 4 show differences between the initial and final latent heat values of both studies despite being done with the same equipment and procedures. The authors also presented some midpoint data about the thermal properties during the experiment, which show a substantial difference on the evolution the latent heat experiences with the cycle time: in Sari and Kaygusuz [40] the latent heat values exponentially decrease with the number of cycles whereas in Sari [33] they do not have a clear pattern as values first decrease but after 600 cycles increase again. Abhat and Malutidis [36] cycled 3 times the lauric acid samples in a Perkin Elmer DSC 2. The only reported data are the latent heat of fusion, the latent heat of crystallization and the melting point after the 3 cycles, but not before the cycling process. No information about the DSC method used is reported in the paper, and the parameter calculation considerations are neither explained, therefore no conclusions about the methodology followed by them can be withdrawn

Myristic acid was studied by Hasan and Sayigh [37], Sari [33], and Sari and Kaygusuz [40]. Studies [33] and [40] used the same setup for the experimental process: a General V4.1C DuPont 2000 DSC, with a heating rate of 10 ºC/min, and a thermostatic chamber setup to cycle the samples. However, results differ a bit and no clear pattern is observed as more energy storage capacity is lost (18%) in study [40] after 910, than in study [33], where the samples were cycled 1200 times and the losses were around 12%. In addition, the midpoint data enclosed in both articles show significant differences on the way the latent heat of fusion decreases with time, as in Sari's [33] it drops a lot before it increases again and in [40] the decrease is mostly constant with the number of cycles. On the other hand, Hasan and Sayigh [37] performed the tests in a Perkin Elmer DSC 2, setting the heating rate to analyse the thermal properties on 10 ºC/min and cycling the samples with a two thermostatic bath setup. Results showed a 14% loss in energy storage capacity, keeping a quite constant melting point that, however, was a bit lower than the melting temperatures experienced in the other two studies.

The same three authors studied palmitic acid with the same experimental setups used in the myristic acid respective studies. Again, studies [33] and [40], done under the same experimental conditions, show higher energy storage capacity loss in the 910 cycles study, 18%, than in the 1200 times cycled study, which is around 13%. They differ on the final latent heat value obtained, lower in study [40], and the diminution tendency experienced by the latent heat. Hasan and Sayigh [37] obtained a lower energy storage capacity loss, 9%, and higher latent heat values cycling the samples 450 times. Abhat and Malutidis [36] also studied this fatty acid. The palmitic acid samples were cycled 5 times in a Perkin Elmer DSC 2 and they enclosed data of the latent heat of fusion, the latent heat of crystallization and the melting point after those 3 cycles, but not before the cycling process. No information about the DSC analysis method neither the parameters calculation is reported in the paper.

Five different authors studied stearic acid thermal properties. Sharma et al. [26] performed their experiments using a Rheometric Scientific Ltd. DSC following a dynamic method and cycling the samples 300 times in an electric hot plate setup. The displayed results show constant melting temperature and a low increase of the latent heat of fusion. These results contrast with the ones obtained under the same experimental conditions in Sharma's later study [27], where the samples where cycled 1500 times and a 21% energy storage capacity loss was experienced by the material. Hasan and Sayigh [37] obtained the highest melting point and latent heat values for stearic acid of all the referenced studies. They cycled the samples using a two thermostatic bath setup and the thermal properties determination was carried out in a Perkin Elmer DSC 2 under a 10 ºC/min heating rate. After 450 cycles the melting temperature remained quite constant but the latent heat of fusion decreased 12%, from 209.9 J/g to 185.3 J/g. The authors also enclosed a table with one mid experimental point after 101 cycles which shows a low decrease on the latent heat value during the first 100 cycles followed by a greater heat storage capacity loss until the final experimental point. The two stearic acid studies that cycled the samples more times were the ones performed by Sari [33] and Sari and Kaygusuz [40]. Both investigations were done under the same experimental conditions, using a thermostatic chamber to cycle the samples and a General V4.1C DuPont 2000 DSC to analyze the thermal properties under a constant heating rate of 10 ºC/min. Results show substantial differences on the latent heat values obtained, as in study [33], after 1200 cycles, the latent heat loss was lower than 2 J/g while in study [40] a 32% loss in the heat storage capacity was experienced after 910 cycles. In both cases some mid experimental point data is attached and the patterns followed by the latent heat of fusion are really different as well: in study [40] latent heat decreases during all the experiment while in study [33] it experiences random increases and decreases during the 1200 cycles. Regarding the melting temperatures, they were quite similar between these two studies. However, both initial and final melting temperatures were considerably lower (15%) than in the other three investigations.

Solé et al. [11] studied the thermal properties of d-mannitol, myo-inositol and galacticol using a Q200 TA Intruments DSC for both cycling and thermal properties measurement. They took the melting temperature as the peak temperature on the DSC curve. The enthalpies were calculated using the area under the peak. They present one mid experimental point for every sugar alcohol study.

2.3 Organic eutectics

The thermal properties of twenty one organic eutectics cycled in thirteen papers of the cited literature are shown in Table 5. Most materials are mixtures of fatty acids, with a few exceptions. The melting points of the eutectics vary from 13 ºC to 64 ºC and the number of cycles ranges from 100 up to 5000.

Just four out of the thirteen different authors present information about the latent heat of fusion and the melting point before and after the cycling tests, and the same papers are the only ones that also show information about the latent heat of crystallization.

50% of the authors do not explain how the melting point and the latent heat of fusion were measured. The others took the phase change temperature as the onset temperature obtained by drawing a line at the point of maximum slope of the leading edge of the DSC peak and extrapolating baseline on the same side as the leading edge of the peak. The latent heat of the phase change was calculated by integration of the area under the peak of the DSC curve.

Ten different DSC instruments were used to obtain the thermal data of these organic eutectics, but only Zhang et al. [32] and Nikolic et al. [39] also used the DSC to cycle the samples. The other studies performed the cycling tests using different equipment and setup configurations.

The eutectic capric acid (65 mol%) + lauric acid (35 mol%) is the only one studied by two different authors. Dimaano and Escoto [50] cycled the eutectic samples 120 times in a thermostatic water bath setup and used a Mettler TA 4000 DSC to measure the thermal properties, while Shilei et al. [53] used an electric hot plate setup to cycle 360 times the samples and a non-specified DSC to determine the thermal properties. Both authors only presented the measures before cycling the samples, and results show that in Shilei et al. [53] the obtained latent heat of fusion was 8% higher than the one obtained by Dimaano and Escoto [50]. The same tendency is observed when comparing the melting points obtained in both studies, as Shilei et al. [53] obtained a 6 ºC higher melting point than Dimaano and Escoto [50] did in their study. So, there already is an important difference on both parameters values even before the cycling stability study.

2.4 Salt hydrates

Cycling of seven salt hydrates has been reported in the literature. The melting temperature of these materials ranges from 8.5 ºC to 111 ºC, and as

Table **6** shows, they have been cycled from 100 to 5650 times.

70% of the authors did not provide data of the final melting point and the latent heat of fusion. Only Fellchenfeld et al. [44] gave information about the latent heat of crystallization and just the 50% of the authors reported how the melting point and the latent heat of fusion were calculated. Indeed, the same methodology was followed by all these 50% more descriptive researchers: the latent heat of fusion was calculated using the area under the peak of the DSC curve, and the melting point was taken as the onset temperature on the same DSC curve.

90% of all the presented studies concerning salt hydrates used a DSC to measure the thermal properties of the different salts. Six different setups were used in these studies to cycle the samples. Porosini [45] was the only author that did not use a DSC in any part of the experimentation as its investigation goal was not to study the heat storage capacity of these salts but to describe the effect of thermal cycling on the PCM physical state.

Five different authors studied calcium chloride hexahydrate heat storage capacity and gave results on its cyclability. Tyagi et al. [48] used a TA Instruments DSC Q-100 to measure the salt's thermal properties and were the only ones that presented information of the melting point and the latent heat of fusion before and after the cycling experiment. Furthermore, they also performed the sample cycling in the DSC. After 1000 cycles, results show constant values for both melting point and latent heat of fusion, hence no heat storage capacity was lost according to them. In addition, the authors also presented mid experimental points to study the pattern that both parameters followed during the whole study, but no clear tendency was seen in neither of the cases; random increases and decreases were experienced by both melting point and latent heat of fusion. Kimura and Kai [60] and Fellchenfeld et al. [44] also cycled 1000 times calcium chloride hexahydrate samples but the results obtained were substantially different from the ones already explained above. Kimura and Kai [60] performed the analysis in a Perkin Elmer DSC 2 but they cycled the salt in a two tube setup with water circulation. The obtained melting point was 6 ºC higher than the achieved in Tyagi et al. [48], and the latent heat of fusion was of 190 J/g, which is 35% more heat storage capacity than Tyagi et al. [48] results. Fellchenfeld et al. [44] measured the thermal properties of the PCM in a Mettler DSC but they performed the cycling using a water bath setup, obtaining the lowest latent heat of fusion of the analyzed studies, 86 J/g, and a melting point of 28 ºC. They also gave the latent heat of crystallization value, which again was really poor, 71 J/g. Porosini [45] did also perform experiments with this salt, however, the study was not focused on determining the heat storage capacity of the salt, hence only information about the initial melting point and the number of cycles is given along with some description of the PCM physical state. Abhat and Malatidis [36] also experimented with this salt, again with a Perkin Elmer DSC 2, but only information about the melting point and the latent heat of fusion after the 18 performed cycles is enclosed in their publication. No method explanation is given by the authors in [36] as well as no specifications on how the parameters were calculated are found in the paper. However, they undertook the experimentation in two different operation modes: with hermetically sealed pans and with non-hermetically sealed or “open” pans. Results show that the salt undergoes decomposition in the tests with open pans, so their recommendation is to use this salt hydrate only in hermetically sealed storage systems.

Glauber’s salt was studied by Marks [59] and Porosini [45]. Despite the analysis methods and the cycling time being different, the same initial melting point was found in both studies. However, the study by Porosini [45] was not focused on determining the heat storage capacity of the salt, hence, no other information but the cycling time and melting point was given in the paper. On the other hand, Marks [59] measured the initial latent heat of fusion and melting point using a calorimeter and cycled the samples in a thermal cycling chamber. The initial melting point and latent heat of fusion results are enclosed in the former Table 5 and the only information reported in the paper after 200 cycles is the latent heat of fusion that decreased until 105 J/g, losing a 56 % of its storage capacity.

El-Sebaii et al. [42,58] did two studies of the magnesium chloride hexahydrate thermal properties after 500 and 1002 cycles, respectively. Both studies used the same equipment and methods: a Heraeus D-6450 Electric Oven was used to cycle the samples and Shimadzu DSC-60 to analyze the thermal properties. In the DSC, the samples were heated at a constant rate of 10 ºC/min between room temperature and 200 ºC and under a constant nitrogen stream. Substantial differences on both initial and final values can be observed, as after 1002 cycles only 6% of the heat storage capacity is lost, while study [42] presented a 46% loss as the latent heat of fusion dropped from 155.11 J/g to 85 J/g in half of the time. In addition, the initial latent heat values of both studies differed 17 ºC. Both measured melting points experienced an important increase with the number of cycles, more remarkable in study [42] as it is of about 11%. The authors also gave midpoint data for both studies from which it can be stated that the latent heat of fusion did not follow any clear pattern in any of the studies, as in both it increases and decreases randomly, with more remarkable variations in study [42], which is the one were a more important latent heat decrease was observed.

2.5 Metal alloys

Table 7 presents the results of the study by Sun et al. [43]on an aluminium-magnesium-zinc alloy that melts at around 450 ºC and was cycled 1000 times. The melting temperature of the alloy corresponds to the onset temperature obtained by drawing a line at the point of maximum slope of the leading edge of the DSC peak and extrapolating the base line on the same side as the leading edge of the peak. The latent heat of fusion was calculated as the area under the peak by numerical integration. They cycled the PCM using a thermostatic chamber and the thermal properties were determined with a Q10-V5.1-Build 191 DSC, performing the analysis in the 298 – 823 ºC range with a constant heating rate of 10 ºC/min under a 80 ml/min nitrogen stream at atmospheric pressure. Results after the 1000 cycles show an 11% loss on the heat storage capacity and a 3 ºC drop on the melting temperature.

2.6 Inorganic eutectics

The cycling methodology and results of seven inorganic eutectics are shown in Table 8. The melting points of the studied eutectics range from 20 ºC to 78 ºC, and all of them were cycled 1000 times.

None of the authors that studied the inorganic eutectics presented in Table 8 gave information about the procedure followed to analyse the thermal properties of the materials. The cycling process and the parameter calculation methods were neither explained in any of the studies.

Kimura and Kai [46] studied the thermal stability of different $CaCl_2$ compounds using the same Perkin Elmer DSC 2. They showed the melting point and heat of fusion values before and after the 1000 cycles.

Nagano et al. [47] did not specify the DSC model used in the study of the $Mg(NO_3)_2$ compound and they presented only the data obtained before cycling the samples. In addition, they also gave the initial value of the latent heat of crystallization. Therefore, conclusions on what happens to these samples when cycled cannot be drawn.

3. Comparison by methodology and equipment

Thermal cycling stability tests are nowadays performed using a wide variety of equipment and setups. Some researchers use DSC, but ovens, thermostatic baths/chambers and electric hot plates are other kind of equipment also employed to perform PCM cycling tests. Other more specific instruments are thermal cyclers. Up to now, thermal cyclers are designed for biomedical applications, though the available temperature range is set from 0 to 100 ºC.

3.1 Equipment

Twenty nine different devices and setups were used to study the thermal stability of fifty eight PCM. Seventeen of these twenty nine devices and setups were differential scanning calorimeters (DSC), while the other 41% were setups exclusively designed to cycle the PCM. Only half of the seventeen DSCs were used for both sample cycling and properties analysing, being the function of the other half to just measure the thermal properties of the already cycled PCM. On the other hand, all the non-DSC equipment displayed in Table 9 were just used to cycle the samples. Regarding these non-DSC setups, it is important to point out here that they have been classified according to the main operational equipment used in each setup; hence, setups included in a same group may have important configuration differences.

The thermal properties of the materials were in all cases measured by DSC except in Porosini's [45] study, were the data was logged directly from the thermostatic chamber setup.

Taking a deeper look on the data presented in Table 9, the huge variety of equipment used to cycle and analyse the PCM does not show any pattern for which one material type is cycled in one concrete setup or DSC and its properties measured in a specific DSC. Hence, no standard is followed by any of the authors when selecting the equipment to conduct their studies, therefore it is reasonable to think that every author studied the thermal stability of each PCM using the equipment he disposed of.

3.2 Cycling method

The cycling method used to analyse the PCM is explained in thirty out of the thirty eight consulted papers. As Table 10 shows, there are two main methods used by the authors: the pyramid method, by which the samples are heated and cooled consecutively, with no isothermal stages amid; and the dynamic method, that stabilizes the PCM samples with isothermal stages before and after every heating and cooling segment. The pyramid method is the most used cycling method, applied by the 83 % of the authors, while the dynamic method is only used in five of the thirty papers that reported the followed cycling procedure. However, five different cool down modes are found within the pyramid method. The cool down at room temperature mode is used in ten papers, which makes it the most used one. The constant rate cooling is used in seven studies, but again different cool down rates in the range 0.7 ºC/min – 10 ºC/min are used by the authors. The other three cooling modes used consisted on cooling in a refrigeration plant, applied in four studies; cooling down at the chamber temperature, which means to cool the sample down in the same chamber where the PCM is heated and it is used by four authors as well; and cooling down in a water bath, mode used by Sun et al. [57] to solidify the Al-34%Mg-6%Zn alloy.

Regarding the dynamic method, three different constant rates are used by the authors in the 5 ºC/min - 10 ºC/min range. In addition, and contrary to the different cool down modes of the pyramid method, both heating and cooling stages have the same flow rate.

Despite of the huge data amount exposed in Table 10, no clear pattern for which a specific material has to be cycled by any of the methods and its different heating/cooling modes is found.

3.3 Analysis method

The analysis methods chosen to measure the PCM thermal properties was reported in twenty five out of the thirty eight consulted papers and are shown in Table 11. The other 34% of the authors did not enclose any information on how the measurements were developed.

The same method types used to cycle the samples are also the ones used by the authors to measure the thermal properties of the PCM: the dynamic method and the pyramid method. The dynamic method is only used in the two different studies by Sharma et al. [26,27], where paraffin wax 53, acetamide and stearic acid were analysed. The pyramid method was the most used, but different heating/cooling rates were applied to analyse the PCM. Zhang et al. [32] measured with a differential scanning calorimeter (DSC) the lauric/palmitc eutectic's thermal properties heating and cooling the samples at a constant rate of 3 ºC/min. Twenty two different PCM of mostly all family types (organic, inorganic, paraffin and eutectics) were analysed under a 5 ºC/min heating/cooling constant rate by eight different authors in eleven studies, which makes it the most common of the heating/cooling rates used. Furthermore, seven different calorimeters were used to do it, being six of them DSC apparatus and the other one a non-specified calorimeter. Calcium chloride hexahydrate's thermal stability was evaluated using a 7 ºC/min constant heating/cooling rate by Tyagi et al. [48], which indeed was the only researcher to select such method. A constant heating/cooling rate of 10 ºC/min was used to measure the thermal properties of eleven different type PCM (organic, inorganic and eutectics, including the only metal alloy studied) in seven different studies where seven different DSC apparatus were used as well. Finally, Sharma et al. [38] analysed fifty time cycled urea using the ramp method as well, but no information on the concrete heating/cooling rate used was reported in their study.

The huge variety on the analysis conditions clearly states that there is no common pattern that specifies the conditions in which each one of the material types has to be studied.

3.4 Number of cycles

Table 12 classifies the PCM as a function of the number of performed cycles in five different cycle ranges, from 0 up to 6000 cycles. As this classification shows, a similar number of PCM is cycled in the first four ranges, being the higher 3001-6000 range the less used one. The clearest fact that can be withdrawn here is that organic PCM (both organic compounds and eutectics) are more often cycled in the low range area, 0 - 500 cycles. However, results clearly show that it does not exist any general standard on the number of cycles a concrete material has to undergo, and that explains, for example, that some organic materials were also cycled more than 3000 times or that $CaCl_2 \cdot 6H_2O$ is found in the lowest (0-100), the middle (501-1000) and the highest (3001-6000) cycle time ranges. Thus, and again, no pattern was found when analysing the cited results by the undergone number of cycles.

4. Conclusions

A review on thermal cycling stability testing has been performed for paraffins, organic materials, salt hydrates, metal alloys, and inorganic eutectics which are commonly used as PCM.

Conclusions are separated in four main aspects according to the main trends to consider when studying the thermal stability of a PCM. These most important parameters are the equipment to cycle the materials, the PCM characterization techniques, the number of cycles to perform and the thermal cycling method.

- Regarding the equipment used and as shown in Table 9, DSC is the most common instrument used to analyse the samples, but just 50 % of the cited authors also use it for cycling. These numbers lead to the first important conclusion that can be withdrawn from this study, which is the lack of a standard that specifies the most useful equipment to perform thermal stability studies.

- The results displayed in the former tables show that DSC is the equipment selected by all authors for PCM thermal characterization. The DSC selection is expected since traditionally DSC has been the most powerful and the most used technique for this purpose. Regarding the analysis conditions, various heating/cooling rates have been used to measure the thermal properties and the 10 ºC/min has been found as the most applied one. That is contrary to the suggestions given by studies [3,64], where low rates are strongly recommended.

- Two main cycling methods, the pyramid and the dynamic, have been found as the most used ones within all the consulted papers. Moreover, the pyramid method is, for far, the most applied one. However, a huge variety of heating rates were used with both methods, being the 5 ºC/min, 7 ºC/min and 10 ºC/min the main chosen ones. This variety of analysis conditions observed leads to conclude that it does not exist any standard methodology to cycle PCM.

- As Table 11 shows, a large variety of cycle ranges has been selected by the different authors and no criteria regarding the PCM family type nor the application are followed in neither case. Thus, and once again, there is a lack of a general standard specifying the number of cycles a specific material has to undergo.

The missing of a general standard for this type of experiments is also observed when different authors or even the same authors perform experiments under the same conditions and obtain different results. No common pattern has been found either when presenting their results. Authors present melting temperature, latent heat of fusion or latent heat of crystallization randomly. Information about the method used, or the equipment is also missing in some of the papers reviewed. Comparing results obtained under the same exact conditions and evaluating them with the same exact methods is the only way by which the results can be analysed and improvements can be applied to obtain better and more accurate parameter values, otherwise, it is impossible to make comparisons if every author has each own criteria when presenting results.

It is mandatory to conclude that a common standard for thermal cycling stability tests would be very appreciated in the scientific community.

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Table 1. Advantages and disadvantages of organic and inorganic PCM.

	Organic	Inorganic
Advantages	Non-corrosive Low or no subcooling Cycling stability	Higher phase change enthalpy Higher conductivity Non-flammable
Disadvantages	Lower phase change enthalpy Lower thermal conductivity Flammable	Subcooling Corrosive Phase separation Lack of cycling stability
Price (€/kg)	6.5-13	3-8

Table 2. Review on the properties listed in the literature.

Reference	Mention on thermal stability	Mention on thermal cycling stability	Mention on thermal cycles	Cycle range	Data availability	Data presented
[12]	+++	+++	+++	1000-5000	+	SEM Images, DSC data,TGA data
[13]	+	++	++	1000-5000	+	NDA
[14]	+++	++	++	NDA	-	NDA
[15]	+	+	+	NDA	-	NDA
[16]	++	+++	+++	5-100	++	SEM Images, DSC data,TGA data
[17]	+	-	-	NDA	-	NDA
[18]	-	-	-	NDA	-	NDA
[19]	++	++	++	NDA	-	NDA
[20]	+	+	+	NDA	-	NDA
[21]	+	++	++	NDA	++	SEM Images, DSC data,TGA data
[22]	+++	+++	+++	1000-5000	+++	SEM Images, DSC data,TGA data
[23]	+++	+++	+++	1000-5000	+++	SEM Images, DSC data,TGA data
[24]	+++	++	++	5-5000	+++	Particle Size distribution
[25]	++	+	+	NDA	-	NDA

*NDA: no data available

Table 3. Melting point and latent heat of thermal cycled paraffins

Sr. nº	**PCM**	**Analytical parameters**							**1)Thermal equipment 2) Cycling equipment**	**Analysis Method**	**Reference**
		Initial melting point (ºC)	**Final melting point (ºC)**	**Initial latent heat of fusion (J/g)**	**Final latent heat of fusion (J/g)**	**Thermal cycles**	**Initial latent heat of crystallization (J/g)**	**Final latent heat of crystallization (J/g)**			
1	Paraffin (70 wt%) + Polypropylene (30 wt%)	44.77	45.52	136.16	116.12	3000	136.59	116.78	1) Setaram DSC 131 2) Electric hot plate setup	5 ºC/min heating rate under a constant stream of argon at a flow rate of 60 mL/min in DSC	Alkan et al. [30]
2	Paraffin ($C_{22}H_{44.1}$) (technical grade)	47.1	46.6	166	163	900	-	-	1) DSC with Mettler TA 3000 system 2) Thermostatic bath setup	No info	Hadjieva et al. [28]
3	Paraffin ($C_{23}H_{48.4}$) (technical grade)	57.1	57.8	220	224	900	-	-	1) DSC with Mettler TA 3000 system 2) Thermostatic bath setup	No info	Hadjieva et al. [28]
4	Paraffin wax 53 (commercial grade)	53	53	184	165	300	-	-	1) Rheometric Scientific Ltd. DSC 2) Electric hot plate setup.	Isothermal–ramp–isothermal method was used in DSC	Sharma et al. [26]
		53	50	184	136	1500	-	-	1) Rheometric Scientific Ltd. DSC	Isothermal–ramp–isothermal method was	Sharma et al. [27]

									2) Electric hot plate setup.	used in DSC	
5	Paraffin wax 58-60	58.27	55	129.8	102	600	-	-	1) Rheometric Scientific Ltd. DSC 2) Oven.	No info	Shukla et al. [29]
6	Paraffin wax 60-62	57.78	59	129.7	109	600	-	-	1) Rheometric Scientific Ltd. DSC 2) Oven.	No info.	Shukla et al. [29]
7	n-Heptadecane/polymethyl methacrylate ($C_{17}H_{36}$)	18.4	18.9	84.7	94.5	5000	18.2	19.1	1) Perkin Elmer Diamond DSC 2) Electric hot plate setup	The heating and cooling rate in DSC was 5 ºC/min in argon atmosphere	Sari et al. [31]
8	Microencapsulated paraffin wax 53 (0.1g)/polyaniline (0.9g)	53.2	53.4	31	30.5	1000	32.6	30.7	1) Thermal box with heater and cooler setup 2) Mettler Toledo 820C DSC	No info	Silakhori et al. [62]
9	Microencapsulated paraffin wax 53 (0.2g)/polyaniline (0.8g)	53.8	53.4	65.1	60.5	1000	66.4	61	1) Thermal box with heater and cooler setup 2) Mettler Toledo 820C DSC	No info	Silakhori et al. [62]

Table 4. Melting point and latent heat of thermal cycled non-paraffin non-eutectic organic materials

Sr. nº	PCM	Analytical parameters							1) Thermal equipment 2) Cycling equipment	Analysis method	Reference
		Initial melting point (ºC)	Final melting point (ºC)	Initial latent heat of fusion (J/g)	Final latent heat of fusion (J/g)	Thermal cycles	Initial latent heat of crystallization (J/g)	Final latent heat of crystallization (J/g)			
1	Acetamide (CH_3CONH_2)	82	81	263	241	300	-	-	1) Rheometric Scientific Ltd. DSC 2) Electric hot plate setup	Isothermal–ramp–isothermal method was used in DSC	Sharma et al. [26]
		82	84	263	260	1500	-	-	1) Rheometric Scientific Ltd. DSC 2) Electric hot plate setup	Isothermal–ramp–isothermal method was used in DSC	Sharma et al. [27]
2	Acetanilide (C_8H_9NO)	113	106	169.4	154	500	-	-	Shimadzu DSC-60	The samples were cycled in the DSC at 10 K/min between ambient temperature and 200 ºC. The analysis was carried out under a constant nitrogen stream of 50mil/min. The samples are heated at a constant rate	El-Sebaii et al. [42]
3	Capric acid (55 wt%) + expanded perlite (45 wt%)	31.80	30.25	98.12	95.54	5000	90.06	90.60	1) Setaram DSC 131 2) Electric hot plate setup	The heating rate in DSC was 5 ºC/min, under a constant stream of argon at atmospheric pressure	Sari and Karaipekli [34]

4	Erythritol	117	119	339	305	1000	-	-	1) Rheometric Scientific Ltd.DSC 2) Oven	No info	Shukla et al. [29]
5	Lauric acid ($C_{11}H_{23}COOH$)	-	43.5	-	169.3	3	-	168.8	Perkin Elmer DSC 2	No info	Abhat and Malatidis [36]
		42.6	41.3	176.6	156.6	1200	-	-	1) General V4.1C DuPont 2000 DSC 2) Thermostatic chamber setup	The heating rate in DSC was 10 ºC/min for all runs	Sari [33]
		42.6	39.5- 44.1	211.6	132.8	910	-	-	1) General V4.1C DuPont 2000 DSC 2) Thermostatic chamber setup	The heating rate in DSC was 10 ºC/min for all runs	Sari and Kaygusuz [40]
6	Methyl palmitate	29	-	215	-	50	214	-	Perkin Elmer DSC - 4	The mean value of at least three samples, recorded by DSC at a heat/cool rate of 5 ºC/min, was taken	Nikolic et al. [39]
7	Methyl stearate	37.8	-	240	-	50	237	-	Perkin Elmer DSC - 4	The mean value of at least three samples, recorded by DSC at a heat/cool rate of 5 ºC/min, was taken	Nikolic et al. [39]
8	Myristic acid ($C_{13}H_{27}COOH$)	50.4	49.8	189.4	163.5	450	-	-	1) Perkin Elmer DSC 2 2) Two thermostatic baths setup	The heating rate in DSC was 10 ºC/min, with a heating range of 10 mcal/s and a scanning range of 290-390 K	Hasan and Sayigh [37]
		52.99	46.21	181.0	159.1	1200	-	-	1) General V4.1C DuPont	The heating rate in DSC was 10	Sari [33]

									2000 DSC 2) Thermostatic chamber setup	ºC/min for all runs	
		53.8	45.3-52.2	192.0	159.1	910	-	-	1) General V4.1C DuPont 2000 DSC 2) Thermostatic chamber setup	The heating rate in DSC was 10 ºC/min for all runs	Sari and Kaygusuz [40]
9	Palmitic acid ($C_{15}H_{31}COOH$)	-	61.2	-	196.1	3	-	197	Perkin Elmer DSC 2	No info	Abhat and Malatidis [36]
		57.8	57.7	201.2	184.4	450	-	-	1) Perkin Elmer DSC 2 2) Two thermostatic baths setup	The heating rate in DSC was 10 ºC/min, with a heating range of 10 mcal/s and a scanning range of 290-390 K	Hasan and Sayigh [37]
		61.31	55.47	197.9	172.4	1200	-	-	1) General V4.1C DuPont 2000 DSC 2) Thermostatic chamber setup	The heating rate in DSC was 10 ºC/min	Sari [33]
		60.9	55.5-62.2	197.9	162.9	910	-	-	1) General V4.1C DuPont 2000 DSC 2) Thermostatic chamber setup	The heating rate In DSC was 10 ºC/min for all runs	Sari and Kaygusuz [40]
10	Palmitic acid (80 wt%) + expanded graphite (20 wt%)	60.88	60.78	148.36	140.38	3000	149.66	139.97	1) Perkin Elmer-Diamond DSC 2) Thermal cycler BIOER TC-25/H model	The heating rate in DSC was 5 ºC/min in a purified argon atmosphere	Sari and Karaipekli [41]

11	Stearic acid ($C_{17}H_{35}COOH$)	65.2	65.9	209.9	185.3	450	-	-	1) Two thermostatic baths setup 2) Perkin Elmer DSC II	The heating rate in DSC was 10 ºC/min, with a heating range of 10 mcal/s and a scanning range of 290-390 K.	Hasan and Sayigh [37]
		62.59	63	154.63	159	300	-	-	1) Rheometric Scientific Ltd. DSC 2) Electric hot plate setup	Isothermal–ramp–isothermal method was used in DSC	Sharma et al. [26]
		54.7	46.83	159.3	157.7	1200	-	-	1) General V4.1C DuPont 2000 DSC 2) Thermostatic chamber setup	The heating rate in DSC was 10 ºC/min for all runs	Sari [33]
		53.8	46.9-50.2	174.6	118.9	910	-	-	1) General V4.1C DuPont 2000 DSC 2) Thermostatic chamber setup	The heating rate in DSC was 10 ºC/min for all runs	Sari and Kaygusuz [40]
		63	64	155	123	1500	-	-	1) Rheometric Scientific Ltd. DSC 2) Electric hot plate setup	Isothermal–ramp–isothermal method was used in DSC	Sharma et al. [27]
12	Urea	133	85.64	239.70	97.91	50			1) Rheometric Scientific Ltd. DSC 2) Electric hot plate setup	Heating at constant rate in DSC	Sharma et al. [38]
13	D-mannitol 99%	150.96	131.92	234.35	99.48	50	224.55	109.5	Q200 TA Instruments DSC	An isothermal-ramp-isothermal method with a heating rate of 10 K/min was used in DSC to cycle the samples under a nitrogen flow of 50 ml/min. 40 µl Tzero aluminium hermetic	Solé et al. [11]

										crucibles were used to contain the samples. The runs in which the thermophysical properties were measured were done at 1 K/min heating rate	
14	Myo-inositol 98%	216.29	221.22	185.25	165.02	100	206.55	176.4	Q200 TA Instruments DSC	An isothermal-ramp-isothermal method with a heating rate of 10 K/min was used in DSC to cycle the samples under a nitrogen flow of 50 ml/min. 40 µl Tzero aluminium hermetic crucibles were used to contain the samples. The runs in which the thermophysical properties were measured were done at 1 K/min heating rate	Solé et al. [11]
15	Galacticol 97%	180.07	-	257.15	-	50	245.65	-	Q200 TA Instruments DSC	An isothermal-ramp-isothermal method with a heating rate of 10 K/min was used in DSC to cycle the samples under a nitrogen flow of 50 ml/min. 40 µl Tzero aluminium hermetic crucibles were used to contain the samples. The runs in which the	Solé et al. [11]

										thermophysical properties were measured were done at 1 K/min heating rate	

Table 5. Melting point and latent heat of thermal cycled organic eutectics

Sr. nº	PCM	Analytical parameters							1) Thermal equipment 2) Cycling equipment	Analysis method	Reference
		Initial melting point (ºC)	Final melting point (ºC)	Initial latent heat of fusion (J/g)	Final latent heat of fusion (J/g)	Thermal cycles	Initial latent heat of crystallization (J/g)	Final latent heat of crystallization (J/g)			
1	Ammonium alum ($NH_4Al(SO_4)_2 \cdot 12H_2O$)(15%) + ammonium nitrate (NH_4NO_3)(85%)	58.5	-	208	-	1100	-	-	1) DSC 2) Calorimeter setup using HDPE balls and thermal bath	5 ºC/min heating rate in DSC	Jotshi et al. [49]
2	Capric acid (65 mol%)+ lauric acid (35 mol%)	13	-	116.76	-	120	-	-	1) Mettler TA 4000 DSC 2) Thermostatic water bath setup	-	Dimaano and Escoto [50]
		19.6	-	126.5	-	360	-	-	1) DSC 2) Electric hot plate setup	-	Shilei et al. [53]
3	Capric acid (73.5 wt%) + myristic acid (26.5 wt%)	22.61	-	154.83	-	5000	156.42	-	1) Setaram DSC 131 2) Two water baths setup	DSC 5 ºC/min constant heating rate, in the temperature range of 10-50 ºC and under a constant stream of argon at atmospheric pressure	Karaipekli et al. [54]

4	Capric acid + (83 wt%) + stearic acid (17 wt%)	24.7	-	178.6	-	5000	-	-	1) Setaram DSC 131 2) Electric hot plate setup	5 ºC/min heating rate was conducted during all DSC measurements	Karaipekli et al. [35]
5	Capric acid (62 wt%) + tetradecanol (38wt%)	18.90	18.12	100.50	96.30	1000	99.70	94.50	1) 204 F1 Phoenix NETZSCH DSC 2) Two thermostatic bath setup with aluminium hollow core slab	DSC: analysis done at a heating rate of 1 ºC/min between 5 and 50 ºC in purified nitrogen atmosphere	Jingyu et al. [63]
6	Caprylic acid (70 wt%) + 1-dodecanol (30 wt%)	6.52	-	171.06	-	120	-	-	1) Perkin Elmer Diamond DSC 2) Thermostatic bath chamber with temperature controller setup	Thermostatic bath chamber: the eutectic mixture was heated above the melting temperature and then cooled below the solidifying temperature DSC: nitrogen was used as the purge gas at a heat flow rate of 20 ml/min. Samples were subjected to three consecutive cooling/heating cycles between -40 and 60 ºC at a scanning rate of 10 ºC/min. The last cooling and heating cycle was used to determine the transition temperatures and enthalpies	Zuo et al. [56]

7	Lauric acid (66 wt%) + myristic acid (34 wt%)	34.2	-	166.8	-	1460	-	-	1) DuPont 2000 DSC 2) Thermostatic chamber with temperature controller	Thermostatic chamber: A thermal cycle consists of heating the PCM above its melting temperature and cooling the PCM at room temperature DSC: the thermal analyses were carried out in the temperature range 0-80 ºC with a heating rate of 5 ºC/min under a constant stream of nitrogen at atmospheric pressure.	Sari [52]
8	Lauric acid (69 wt%) + palmitic acid (31 wt%)	35.2	34.8	166.3	168.8	1460	-	-	1) DuPont 2000 DSC 2) Thermostatic chamber with temperature controller	Thermostatic chamber: A thermal cycle consists of heating the PCM above its melting temperature and cooling the PCM at room temperature DSC: the thermal analyses were carried out in the temperature range 0-80 ºC with a heating rate of 5 ºC/min under a constant stream of nitrogen at atmospheric pressure	Sari [52]
9	Lauric acid (75.5 wt%) + stearic acid (24.5 wt%)	37	-	182.7	-	360	-	-	1) DuPont 2000 DSC 2) Thermostatic chamber with	Thermostatic chamber: A thermal cycle consists of heating the PCM above its melting temperature and cooling the PCM at room temperature	Sari et al. [51]

									temperature controller	DSC: the thermal analyses were carried out in the temperature range 0-80 ºC with a heating rate of 5 ºC/min under a constant stream of nitrogen at atmospheric pressure	
10	Lauric acid (77.05 wt%) + palmitic acid (22.95 wt%)	33.09	32.92	150.6	165.69	100	-	-	Perkin Elmer DSC7	DSC: scanning rate of 3 ºC/min in the temperature range of 30-70 ºC	Zhang et al. [32]
11	Methyl stearate (86 wt%) + methyl palmitate (14 wt%)	23.9	-	220	-	50	220	-	Perkin Elmer DSC4	DSC: heat/cool rate of 5 ºC/min	Nikolic et al. [39]
12	Methyl stearate (91 wt%) + cetyl palmitate (9 wt%)	28.2	-	189	-	50	185	-	Perkin Elmer DSC4	DSC: heat/cool rate of 5 ºC/min	Nikolic et al. [39]
13	Methyl stearate (91 wt%) + cetyl stearate (9 wt%)	22.2	-	180	-	50	175	-	Perkin Elmer DSC4	DSC: heat/cool rate of 5 ºC/min	Nikolic et al. [39]
14	Myristic acid (58 wt%) + palmitic acid (42 wt%)	42.6	-	169.7	-	360	-	-	1) DuPont 2000 DSC 2) Thermostatic chamber with temperature controller	Thermostatic chamber: A thermal cycle consists of heating the PCM above its melting temperature and cooling the PCM at room temperature DSC: the thermal analyses were	Sari et al. [51]

										carried out in the temperature range 0-80 ºC with a heating rate of 5 ºC/min under a constant stream of nitrogen at atmospheric pressure	
15	Myristic acid (64 wt%) + stearic acid (36 wt%)	44.1	-	182.4	-	1460	-	-	1) DuPont 2000 DSC 2) Thermostatic chamber with temperature controller	Thermostatic chamber: A thermal cycle consists of heating the PCM above its melting temperature and cooling the PCM at room temperature DSC: the thermal analyses were carried out in the temperature range 0-80 ºC with a heating rate of 5 ºC/min under a constant stream of nitrogen at atmospheric pressure	Sari [52]
16	Myristic acid + glycerol	31.96	31.22	154.3	151.8	1000	148.8	153.4	1) PerkinElmer JADE DSC 2) Thermostatic chamber with temperature controller	Thermostatic chamber: A thermal cycle consists of heating the PCM above its melting temperature and cooling the PCM at room temperature DSC: the analyses were performed at 5 ºC/min of constant heating rate, in the temperature range 5-80 ºC, under a constant stream of argon at atmospheric pressure	Sari et al. [55]

17	Palmitic acid (64.2 wt%) + stearic acid (35.8 wt%)	52.3	-	181.7	-	360	-	-	1) DuPont 2000 DSC 2) Thermostatic chamber with temperature controller	Thermostatic chamber: A thermal cycle consists of heating the PCM above its melting temperature and cooling the PCM at room temperature DSC: the thermal analyses were carried out in the temperature range 0-80 ºC with a heating rate of 5 ºC/min under a constant stream of nitrogen at atmospheric pressure	Sari et al. [51]
18	Palmitic acid + glycerol	58.50	57.45	185.9	175.8	1000	151.7	147.5	1) PerkinElmer JADE DSC 2) Thermostatic chamber with temperature controller	Thermostatic chamber: A thermal cycle consists of heating the PCM above its melting temperature and cooling the PCM at room temperature DSC: the analyses were performed at 5 ºC/min of constant heating rate, in the temperature range 5-80 ºC, under a constant stream of argon at atmospheric pressure	Sari et al. [55]
19	Stearic acid + glycerol	63.45	62.83	149.4	152.8	1000	64.58	60.06	1) PerkinElmer JADE DSC 2) Thermostatic chamber with	Thermostatic chamber: A thermal cycle consists of heating the PCM above its melting temperature and cooling the PCM at room temperature	Sari et al. [55]

									temperature controller	DSC: the analyses were performed at 5 ºC/min of constant heating rate, in the temperature range 5-80 ºC, under a constant stream of argon at atmospheric pressure	
20	Tetradodecanol (53.60 wt%) + lauric acid (46.40 wt%)	24.53	23.22	90.20	85.70	1000	88.70	85.10	1) 204 F1 Phoenix NETZSCH DSC 2) Two thermostatic bath setup with aluminium hollow core slab	DSC: analysis done at a heating rate of 1 ºC/min between 5 and 50 ºC in purified nitrogen atmosphere	Jingyu et al. [63]
21	Tetradodecanol (71.84 wt%) + myristic acid (28.16 wt%)	33.15	32.65	128.60	123.50	1000	125.70	121.70	1) 204 F1 Phoenix NETZSCH DSC 2) Two thermostatic bath setup	DSC: analysis done at a heating rate of 1 ºC/min between 5 and 50 ºC in purified nitrogen atmosphere	Jingyu et al. [63]

Table 6. Melting point and latent heat of thermal cycled salt hydrates

Sr. nº	PCM	Analytical parameters							1) Thermal equipment 2) Cycling equipment	Analysis method	Reference
		Initial melting point (ºC)	Final melting point (ºC)	Initial latent heat of fusion (J/g)	Final latent heat of fusion (J/g)	Thermal cycles	Initial latent heat of crystallization (J/g)	Final latent heat of crystallization (J/g)			
1	Calcium chloride hexahydrate ($CaCl_2 \cdot 6H_2O$)	29.8	-	190.8	-	1000	-	-	1) Perkin Elmer DSC 2 2) Heat Exchange by water circulation in a two tube setup	No info	Kimura and Kai [60]
		28	-	86	-	1000	71	-	1) Mettler DSC 2) Water bath setup	No info	Fellchenfeld et al. [44]
		27	-	-	-	5650	-	-	Thermostatic chamber	Stainless steel capsules were filled with 50 g of samples and introduced in a thermostatic chamber where they were cycled and from where all data was logged	Porosini [45]
		23.26	23.26	125.4	125.4	1000	-	-	TA Instruments DSC Q-100	Cycling with DSC at a constant rate of 7 ºC/min between -10 and 60 ºC	Tyagi et al. [48]
		-	29	-	160	18	-	-	Perkin Elmer DSC 2	No info	Abhat and

											Malatidis [36]
2	Glauber’s salt ($Na_2SO_4 \cdot 10H_2O$)	32.4	-	238	-	320	-	-	1) Calorimeter 2) Thermal cycling chamber	No DSC used. The paper explains the calorimetric measure method	Marks [59]
		32	-	-	-	5650	-	-	Thermostatic chamber	Stainless steel capsules were filled with 50 g of samples and introduced in a thermostatic chamber where they were cycled and from where all data was logged	Porosini [45]
3	Magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$)	111.5	124.12	155.11	85	500	-	-	1) Shimadzu DSC-60 2) Heraeus D-6450 Electric Oven	The samples were cycled in the DSC at 10 K/min between ambient temperature and 200 ºC. The analysis was carried out under a constant nitrogen stream of 50mil/min. The samples are heated at a constant rate	El-Sebaii et al. [42]
		110.8	115.39	138	130.28	1002	-	-	1) Shimadzu DSC-60 2) Heraeus D-6450 Electric Oven	The samples were cycled in the DSC at 10 K/min between ambient temperature and 200 ºC. The analysis was carried out under a constant nitrogen stream of 50mil/min. The samples are heated at a	El-Sebaii et al. [58]

										constant rate	
4	$Na_2SO_4 \cdot 1/2NaCl \cdot 10H_2O$	20	-	-	-	5650	-	-	Thermostatic chamber	Stainless steel capsules were filled with 50 g of samples and introduced in a thermostatic chamber where they were cycled and from where all data was logged	Porosini [45]
5	$NaOH \cdot 3,5H_2O$	15	-	-	-	5650	-	-	Thermostatic chamber	Stainless steel capsules were filled with 50 g of samples and introduced in a thermostatic chamber where they were cycled and from where all data was logged	Porosini [45]
6	Sodium acetate trihydrate ($NaCH_3COO \cdot 3H_2O$)	58	-	230	-	500	-	-	1) Calorimeter 2) Thermal bath	The stainless steel vessel that contained the sample was put into a water bath a consecutively heated and cooled at a rate of 5 ºC/min. Calorimetric measurements were performed to measure the thermal properties.	Wada et al. [61]
7	Trichlorofluoromethane heptadecahydrate ($CCl_3F \cdot 17H_2O$)	8.5	-	274	-	100	-	-	1) Perkin Elmer DSC 2 2) Glass beaker with water jacket	No info	Kimura and Kai [43]

Table 7. Melting point and latent heat of thermal cycled metallic alloys

Sr. nº	PCM	Initial melting point (ºC)	Final melting point (ºC)	Initial latent heat of fusion (J/g)	Final latent heat of fusion (J/g)	Thermal cycles	Initial latent heat of crystallization (J/g)	Final latent heat of crystallization (J/g)	1) Thermal equipment 2) Cycling equipment	Analysis method	Reference
1	Al-34%Mg-6%Zn alloy	450.31	447.25	329.1	292.96	1000	-	-	1) Q10-V5.1-Build191 DSC 2) Thermostatic chamber	The DSC thermal analyses were performed in the temperature range of 298–823 K with a heating rate of 10 K/min and under 80 ml/min of nitrogen at atmospheric pressure	Sun et al. [57]

Table 8. Melting point and latent heat of thermal cycled inorganic eutectics

Sr. nº	PCM	Analytical parameters							1) Thermal equipment 2) Cycling equipment	Analysis method	Reference
		Initial melting point (ºC)	Final melting point (ºC)	Initial latent heat of fusion (J/g)	Final latent heat of fusion (J/g)	Thermal cycles	Initial latent heat of crystallization (J/g)	Final latent heat of crystallization (J/g)			
1	$CaCl_2 \cdot 6H_2O$ (80 mol%) + $CaBr_2 \cdot 6H_2O$ (20 mol%)	20	21	117	120	1000	-	-	Perkin Elmer DSC 2	No info.	Kimura and Kai [46]
2	$CaCl_2 \cdot 6H_2O$ (93 wt%) + $Ca(NO_3)_2 \cdot 4H_2O$ (5 wt%) + $Mg(NO_3)_2 \cdot 6H_2O$ (2 wt%)	24	23	125	128	1000	-	-	Perkin Elmer DSC 2	No info.	Kimura and Kai [46]
3	$CaCl_2 \cdot 6H_2O$ (96 wt%) + KNO_3 (2 wt%) +KBr (2 wt%)	23	23	138	139	1000	-	-	Perkin Elmer DSC 2	No info.	Kimura and Kai [46]
4	$CaCl_2 \cdot 6H_2O$ (96 wt%) + NH_4NO_3 (2 wt%) + NH_4Br (2 wt%)	20	19	141	141	1000	-	-	Perkin Elmer DSC 2	No info.	Kimura and Kai [46]
5	$Mg(NO_3)_2 \cdot 6H_2O$ (93 wt%) + $MgCl_2 \cdot 6H_2O$ (7 wt%)	78	-	152.4	-	1000	77.2	-	DSC	No info.	Nagano et al. [47]

Table 9. List of all the equipment and setups used for analysis and cycling

Equipment	Model	Materials	Authors
Setaram DSC	131	Paraffin 70% + PP 30%	Alkan et al. [30]
		Capric acid (55 wt%) + expanded perlite (45 wt%)	Sari and Karaipekli [34]
		Capric acid (73.5 wt%) + myristic acid (26.5 wt%)	Karaipekli et al. [54]
		Capric acid + (83 wt%) + stearic acid (17 wt%)	Karaipekli et al. [35]
Mettler TA DSC	4000	Capric acid (65 mol%) + lauric acid (35 mol%)	Dimaano and Escoto [50]
Mettler TA DSC	-	Paraffin ($C_{22}H_{44.1}$)	Hadjieva et al. [28]
		Paraffin ($C_{23}H_{48.4}$)	Hadjieva et al. [28]
		$CaCl_2 \cdot 6H_2O$	Fellchenfeld et al. [44]
Rheometric Scientific Ltd. DSC	-	Paraffin wax 53	Sharma et al. [26]
			Sharma et al. [27]
		Paraffin wax 58-60	Shukla et al. [29]
		Paraffin wax 60-62	Shukla et al. [29]
		Acetamide	Sharma et al. [26]
			Sharma et al. [27]
		Erythritol	Shukla et al. [29]
		Stearic acid	Sharma et al. [26]
			Sharma et al. [27]
		Urea	Sharma et al. [38]
Perkin Elmer DSC	Diamond	$C_{17}H_{36}$	Sari et al. [31]
		Palmitic acid (80 wt%) + expanded graphite (20 wt%)	Sari and Karaipekli [41]
		Caprylic acid (70 wt%) + 1-dodecanol (30 wt%)	Zuo et al. [56]

Perkin Elmer DSC	Jade	Myristic acid + glycerol	Sari et al. [55]
		Palmitic acid + glycerol	Sari et al. [55]
		Stearic acid + glycerol	Sari et al. [55]
Perkin Elmer DSC	7	Lauric acid (77.05 wt%) + palmitic acid (22.95 wt%)	Zhang et al. [32]
Perkin Elmer DSC	4	Methyl palmitate	Nikolic et al. [39]
		Methyl stearate	Nikolic et al. [39]
		Methyl stearate (86 wt%) + methyl palmitate (14 wt%)	Nikolic et al. [39]
		Methyl stearate (91 wt%) + cetyl palmitate (9 wt%)	Nikolic et al. [39]
		Methyl stearate (91 wt%) + cetyl stearate (9 wt%)	Nikolic et al. [39]
Perkin Elmer DSC	2	Myristic acid	Hasan and Sayigh [37]
		Palmitic acid	Abhat and Malatidis [36]
		Stearic acid	Hasan and Sayigh [37]
		$CaCl_2 \cdot 6H_2O$	Abhat and Malatidis [36]
		$CCl_3F \cdot 17H_2O$	Kimura and Kai [43]
		$CaCl_2 \cdot 6H_2O$ compounds	Kimura and Kai [46]
		Lauric acid	Abhat and Malatidis [36]
Shimadzu DSC	60	Acetanilide	El-Sebaii et al. [42]
		$MgCl_2 \cdot 6H_2O$	El-Sebaii et al. [58]
DuPont DSC	General V4.1C 2000	Lauric acid	Sari [33]
			Sari and Kaygusuz [40]
		Myristic acid	Sari [33]
			Sari and Kaygusuz [40]
		Palmitic acid	Sari [33]

			Sari and Kaygusuz [40]
		Stearic acid	Sari [33]
			Sari and Kaygusuz [40]
DuPont DSC	2000	Lauric acid (66 wt%) + myristic acid (34 wt%)	Sari [52]
		Lauric acid (69 wt%) + palmitic acid (31 wt%)	Sari [52]
		Lauric acid (75.5 wt%) + stearic acid (24.5 wt%)	Sari et al. [51]
		Myristic acid (58 wt%) + palmitic acid (42 wt%)	Sari et al. [51]
		Myristic acid (64 wt%) + stearic acid (36 wt%)	Sari [52]
		Palmitic acid (64.2 wt%) + stearic acid (35.8 wt%)	Sari et al. [51]
TA Instruments DSC	Q2000	D-mannitol 99%	Solé et al. [51]
		Myo-inositol 98%	Solé et al. [51]
		Galacticol 97%	Solé et al. [51]
Mettler Toledo DSC	820C	Microencapsulated paraffin wax 53 (0.1g)/polyaniline (0.9g)	Silakhori et al. [52]
		Microencapsulated paraffin wax 53 (0.2g)/polyaniline (0.8g)	Silakhori et al. [52]
TA Instruments DSC	Q100	$CaCl_2 \cdot 6H_2O$	Tyagi et al. [48]
Build DSC	Q10-V5.1 191	Al-34%Mg-6%Zn alloy	Sun et al. [57]
NETZSCH DSC	204 F1 Phoenix	Capric acid (62 wt%) + tetradecanol (38wt%)	Jingyu et al. [63]
		Tetradodecanol (53.60 wt%) + lauric acid (46.40 wt%)	Jingyu et al. [63]
		Tetradodecanol (71.84 wt%) + myristic acid (28.16 wt%)	Jingyu et al. [63]
Thermal cycler BIOER	TC-25/H	Palmitic acid (80 wt%) + expanded graphite (20 wt%)	Sari and Karaipekli [41]
Thermal cycling chamber	-	Glauber’s salt	Marks [59]
Thermostatic chamber setup	-	Lauric acid	Sari [33]
			Sari and Kaygusuz [40]

		Myristic acid	Sari [33]
			Sari and Kaygusuz [40]
		Palmitic acid	Sari [33]
			Sari and Kaygusuz [40]
		Stearic acid	Sari [33]
			Sari and Kaygusuz [40]
		$CaCl_2 \cdot 6H_2O$	Fellchenfeld et al. [44]
			Porosini [45]
		Glauber's salt	Porosini [45]
		$Na_2SO_4 \cdot 1/2NaCl \cdot 10H_2O$	Porosini [45]
		$NaOH \cdot 3{,}5H_2O$	Porosini [45]
		Al-34%Mg-6%Zn alloy	Sun et al. [57]
		Caprylic acid (70 wt%) + 1-dodecanol (30 wt%)	Zuo et al. [56]
		Lauric acid (66 wt%) + myristic acid (34 wt%)	Sari [52]
		Lauric acid (69 wt%) + palmitic acid (31 wt%)	Sari [52]
		Lauric acid (75.5 wt%) + stearic acid (24.5 wt%)	Sari et al. [51]
		Myristic acid (58 wt%) + palmitic acid (42 wt%)	Sari et al. [51]
		Myristic acid (64 wt%) + stearic acid (36 wt%)	Sari [52]
		Myristic acid + glycerol	Sari et al. [55]
		Palmitic acid (64.2 wt%) + stearic acid (35.8 wt%)	Sari et al. [51]
		Palmitic acid + glycerol	Sari et al. [55]
		Stearic acid + glycerol	Sari et al. [55]
Thermal bath setup	-	Capric acid (65 mol%)+ lauric acid (35 mol%)	Dimaano and Escoto [50]

		$NaCH_3COO \cdot 3H_2O$	Wada et al. [61]
		Paraffin ($C_{22}H_{44.1}$)	Hadjieva et al. [28]
		Paraffin ($C_{23}H_{48.4}$)	Hadjieva et al. [28]
		Paraffin (70 wt%) + PP (30 wt%)	Alkan et al. [30]
		$C_{17}H_{36}$	Sari et al. [31]
Two thermostatic bath setup	-	Myristic acid	Hasan and Sayigh [37]
		Palmitic acid	Hasan and Sayigh [37]
		Stearic acid	Hasan and Sayigh [37]
		Capric acid (73.5 wt%) + myristic acid (26.5 wt%)	Karaipekli et al. [54]
		Capric acid (62 wt%) + tetradecanol (38wt%)	Jingyu et al. [63]
		Tetradodecanol (53.60 wt%) + lauric acid (46.40 wt%)	Jingyu et al. [63]
		Tetradodecanol (71.84 wt%) + myristic acid (28.16 wt%)	Jingyu et al. [63]
Glass beaker with water jacket circulation	-	$CCl_3F \cdot 17H_2O$	Kimura and Kai [43]
Electric hot plate setup	-	Paraffin wax 53	Sharma et al. [26]
			Sharma et al. [27]
		Acetamide	Sharma et al. [26]
			Sharma et al. [27]
		Stearic acid	Sharma et al. [26]
			Sharma et al. [27]
		Urea	Sharma et al. [38]
		Capric acid (65 mol%)+ lauric acid (35 mol%)	Shilei et al. [53]
		Capric acid + (83 wt%) + stearic acid (17 wt%)	Karaipekli et al. [35]
		Capric acid (55 wt%) + expanded perlite (45 wt%)	Sari and Karaipekli [34]

Two tube with water circulation setup	-	$CaCl_2 \cdot 6H_2O$	Kimura and Kai [60]
Thermal box with heater and cooler setup	-	Microencapsulated paraffin wax 53 (0.1g)/polyaniline (0.9g)	Silakhori et al. [62]
		Microencapsulated paraffin wax 53 (0.2g)/polyaniline (0.8g)	Silakhori et al. [62]
Oven	Non specified	Paraffin wax 58-60	Shukla et al. [29]
		Paraffin wax 60-62	Shukla et al. [29]
		Erythritol	Shukla et al. [29]
Oven	Heraeus D-6450	$MgCl_2 \cdot 6H_2O$	El-Sebaii et al. [42]
			El-Sebaii et al. [58]
Calorimeter + thermal bath	-	$(NH_4Al(SO_4)_2 \cdot 12H_2O)(15\%) + (NH_4NO_3)(85\%)$	Jotshi et al. [49]

Table 10. List of the different cycling methods used by the authors

Cycling method		**Materials**	**Equipment**	**References**
Pyramid method Λ	Cool down in refrigeration plant	Paraffin (70 wt%) + PP (30 wt%)	Electric hot plate setup	Alkan et al. [30]
		$C_{17}H_{36}$	Electric hot plate setup	Sari et al. [31]
		Capric acid (55 wt%) + expanded perlite (45 wt%)	Electric hot plate setup	Sari and Karaipekli [34]
		Capric acid (65 mol%)+ lauric acid (35 mol%)	Electric hot plate setup	Shilei et al. [53]
	Cool down at room temperature	Paraffin wax 53	Electric hot plate setup	Sharma et al. [26]
			Electric hot plate setup	Sharma et al. [27]
		Acetamide	Electric hot plate setup	Sharma et al. [26]
			Electric hot plate setup	Sharma et al. [27]
		Paraffin wax 58-60	Oven	Shukla et al. [29]
		Paraffin wax 60-62	Oven	Shukla et al. [29]
		Erythritol	Oven	Shukla et al. [29]
		Lauric acid	Thermostatic chamber setup	Sari [33]
			Thermostatic chamber setup	Sari and Kaygusuz [40]
		Myristic acid	Thermostatic chamber setup	Sari [33]
			Thermostatic chamber setup	Sari and Kaygusuz [40]
		Palmitic acid	Thermostatic chamber setup	Sari [33]
			Thermostatic chamber setup	Sari and Kaygusuz [40]
		Stearic acid	Electric hot plate setup	Sharma et al. [26]
			Thermostatic chamber setup	Sari [33]
			Thermostatic chamber setup	Sari and Kaygusuz [40]
			Electric hot plate setup	Sharma et al. [27]

			Urea	Electric hot plate setup	Sharma et al. [38]
			Capric acid + (83 wt%) + stearic acid (17 wt%)	Electric hot plate setup	Karaipekli et al. [35]
			Lauric acid (66 wt%) + myristic acid (34 wt%)	Thermostatic chamber setup	Sari [52]
			Lauric acid (69 wt%) + palmitic acid (31 wt%)	Thermostatic chamber setup	Sari [52]
			Myristic acid (64 wt%) + stearic acid (36 wt%)	Thermostatic chamber setup	Sari [52]
			Lauric acid (75.5 wt%) + stearic acid (24.5 wt%)	Thermostatic chamber setup	Sari [51]
			Myristic acid (58 wt%) + palmitic acid (42 wt%)	Thermostatic chamber setup	Sari [51]
			Myristic acid (64 wt%) + stearic acid (36 wt%)	Thermostatic chamber setup	Sari [52]
			Myristic acid + glycerol	Thermostatic chamber setup	Sari [55]
			Palmitic acid + glycerol	Thermostatic chamber setup	Sari [55]
			Stearic acid + glycerol	Thermostatic chamber setup	Sari [55]
	Cool down in water bath		Myristic acid	Two thermostatic bath setup	Hasan and Sayigh [37]
			Stearic acid	Two thermostatic bath setup	Hasan and Sayigh [37]
			$(NH_4Al(SO_4)_2 \cdot 12H_2O)$(15%) + (NH_4NO_3)(85%)	Calorimeter + thermal bath	Jotshi et al. [49]
			Capric acid (73.5 wt%) + myristic acid (26.5 wt%)	Two thermostatic bath setup	Karaipekli et al. [54]
	Cool down at chamber temperature		Al-34%Mg-6%Zn alloy	Thermostatic chamber setup	Sun et al. [57]
	Constant heating/cooling rate	0.7 ºC/min	$CaCl_2 \cdot 6H_2O$	Thermostatic chamber setup	Feilchenfeld et al. [44]
		3 ºC/min	Lauric acid (77.05 wt%) + palmitic acid (22.95 wt%)	Perkin Elmer DSC 7	Zhang et al. [32]
		1.2 ºC/min	Paraffin ($C_{22}H_{44.1}$)	Thermostatic bath setup	Hadjieva et al. [28]
			Paraffin ($C_{23}H_{48.4}$)	Thermostatic bath setup	Hadjieva et al. [28]
		5 ºC/min	Methyl palmitate	Perkin Elmer DSC 4	Nikolic et al. [39]

			Methyl stearate	Perkin Elmer DSC 4	Nikolic et al. [39]
			Methyl stearate (86 wt%) + methyl palmitate (14 wt%)	Perkin Elmer DSC 4	Nikolic et al. [39]
			Methyl stearate (91 wt%) + cetyl palmitate (9 wt%)	Perkin Elmer DSC 4	Nikolic et al. [39]
			Methyl stearate (91 wt%) + cetyl stearate (9 wt%)	Perkin Elmer DSC 4	Nikolic et al. [39]
		10 ºC/min	Acetanilide	Shimadzu DSC-60	El-Sebaii et al. [42]
		Non specified	Glauber's salt	Thermal cycling chamber	Marks [59]
			$MgCl_2 \cdot 6H_2O$	Heraeus D-6450 Electric Oven	El-Sebaii et al. [42]
				Heraeus D-6450 Electric Oven	El-Sebaii et al. [58]
Dynamic method _/‾_	Constant heating/cooling rate	5 ºC/min	Sodium acetate trihydrate	Thermal bath setup	Wada et al. [61]
		7 ºC/min	$CaCl_2 \cdot 6H_2O$	TA Instruments DSC Q-100	Tyagi et al. [48]
		10 ºC/min	D-mannitol 99%	Q200 TA Instruments DSC	Solé et al. [11]
			Myo-inositol 98%	Q200 TA Instruments DSC	Solé et al. [11]
			Galacticol 97%	Q200 TA Instruments DSC	Solé et al. [11]
			$CaCl_2 \cdot 6H_2O$	Two tube with water circulation setup	Kimura and Kai [60]
		Non specified	Capric acid (65 mol%)+ lauric acid (35 mol%)	Thermal bath setup	Dimaano and Escoto [50]

Table 11. List of the different analysis methods used by the authors

Analysis Method		Equipment	Materials	References
Pyramid method	Heating /cooling at 3 ºC/min constant rate	Perkin Elmer DSC 7	Lauric acid (77.05 wt%) + palmitic acid (22.95 wt%)	Zhang et al. [32]
	Heating/cooling at 5 ºC/min constant rate	Setaram DSC 131	Paraffin (70 wt%) + PP (30 wt	Alkan et al. [30]
		Perkin Elmer Jade DSC	$C_{17}H_{36}$	Sari et al. [31]
		Setaram DSC 131	Capric acid (55 wt%) + expanded perlite (45 wt%)	Sari and Karaipekli [34]
		Perkin Elmer DSC 4	Methyl palmitate	Nikolic et al. [39]
		Perkin Elmer DSC 4	Methyl stearate	Nikolic et al. [39]
		Perkin Elmer Diamond DSC	Palmitic acid (80 wt%) + expanded graphite (20 wt%)	Sari and Karaipekli [41]
		Calorimeter	$NaCH_3COO \cdot 3H_2O$	Wada et al. [61]
		DSC	$(NH_4Al(SO_4)_2 \cdot 12H_2O)$(15%) + (NH_4NO_3)(85%)	Jotshi et al. [49]
		Setaram DSC 131	Capric acid (73.5 wt%) + myristic acid (26.5 wt%)	Karaipekli et al. [54]
		Setaram DSC 131	Capric acid + (83 wt%) + stearic acid (17 wt%)	Karaipekli et al. [35]
		DuPont 2000 DSC	Lauric acid (66 wt%) + myristic acid (34 wt%)	Sari [52]
		DuPont 2000 DSC	Lauric acid (69 wt%) + palmitic acid (31 wt%)	Sari et al. [51]
		DuPont 2000 DSC	Lauric acid (75.5 wt%) + stearic acid (24.5 wt%)	Sari et al. [51]
		Perkin Elmer DSC 4	Methyl stearate (86 wt%) + methyl palmitate (14 wt%)	Nikolic et al. [39]
		Perkin Elmer DSC 4	Methyl stearate (91 wt%) + cetyl palmitate (9 wt%)	Nikolic et al. [39]
		Perkin Elmer DSC 4	Methyl stearate (91 wt%) + cetyl stearate (9 wt%)	Nikolic et al. [39]
		DuPont 2000 DSC	Myristic acid (58 wt%) + palmitic acid (42 wt%)	Sari et al. [51]
		DuPont 2000 DSC	Myristic acid (64 wt%) + stearic acid (36 wt%)	Sari et al. [52]
		Perkin Elmer Jade DSC	Myristic acid + glycerol	Sari et al. [55]

		DuPont 2000 DSC	Palmitic acid (64.2 wt%) + stearic acid (35.8 wt%)	Sari et al. [51]
		Perkin Elmer Jade DSC	Palmitic acid + glycerol	Sari et al. [55]
		Perkin Elmer Jade DSC	Stearic acid + glycerol	Sari et al. [55]
	Heating/cooling at 7 ºC/min constant rate	TA Instruments DSC	$CaCl_2 \cdot 6H_2O$	Tyagi et al. [48]
	Heating/cooling at 10 ºC/min constant rate	Shimadzu DSC-60	Acetanilide	El-Sebaii et al. [42]
		General V4.1C DuPont 2000 DSC	Lauric acid	Sari [33]
				Sari and Kaygusuz [40]
		Perkin Elmer DSC 2	Myristic acid	Hasan and Sayigh [37]
		General V4.1C DuPont 2000 DSC		Sari [33]
		General V4.1C DuPont 2000 DSC		Sari and Kaygusuz [40]
		Perkin Elmer DSC 2	Palmitic acid	Hasan and Sayigh [37]
		General V4.1C DuPont 2000 DSC		Sari [33]
		General V4.1C DuPont 2000 DSC		Sari and Kaygusuz [40]
		Perkin Elmer DSC 2	Stearic acid	Hasan and Sayigh [37]
		General V4.1C DuPont 2000 DSC		Sari [33]
		General V4.1C DuPont 2000 DSC		Sari and Kaygusuz [40]
		Q2000 TA Instruments DSC	D-mannitol 99%	Solé et al. [11]

		Q2000 TA Instruments DSC	Myo-inositol 98%	Solé et al. [11]
		Q2000 TA Instruments DSC	Galacticol 97%,	Solé et al. [11]
		Shimadzu DSC-60	$MgCl_2 \cdot 6H_2O$,	El-Sebaii et al. [42]
				El-Sebaii et al. [58]
		Q10-V5.1-Build191 DSC	Al-34%Mg-6%Zn alloy	Sun et al. [57]
		Perkin Elmer Diamond DSC	Caprylic acid (70 wt%) + 1-dodecanol (30 wt%)	Zuo et al. [56]
	Non specified constant rate	Rheometric Scientific Ltd. DSC	Urea	Sharma et al. [38]
Dynamic method	No specifications	Rheometric Scientific Ltd. DSC	Paraffin wax 53	Sharma et al. [26]
			Paraffin wax 53	Sharma et al. [27]
			Acetamide	Sharma et al. [26]
			Acetamide	Sharma et al. [27]
			Stearic acid	Sharma et al. [26]
			Stearic acid	Sharma et al. [27]

Table 12. Materials classified by number of cycles range

Cycles	Materials	Equipment	Authors
0-100	Lauric acid (77.05 wt%) + palmitic acid (22.95 wt%)	Perkin Elmer DSC 7	Zhang et al. [32]
	Methyl palmitate	Perkin Elmer DSC 4	Nikolic et al. [39]
	Methyl stearate	Perkin Elmer DSC 4	Nikolic et al. [39]
	Urea	Electric hot plate setup	Sharma et al. [38]
	D-mannitol 99%,	Q200 TA Instruments DSC	Solé et al. [11]
	Myo-inositol 98%	Q200 TA Instruments DSC	Solé et al. [11]
	Galacticol 97%	Q200 TA Instruments DSC	Solé et al. [11]
	$CCl_3F \cdot 17H_2O$	Glass beaker with water jacket	Kimura and Kai [43]
	Methyl stearate (86 wt%) + methyl palmitate (14 wt%)	Perkin Elmer DSC 4	Nikolic et al. [39]
	Methyl stearate (91 wt%) + cetyl palmitate (9 wt%)	Perkin Elmer DSC 4	Nikolic et al. [39]
	Lauric acid	Perkin Elmer DSC 2	Abhat and Malatidis [36]
	Palmitic acid	Perkin Elmer DSC 2	Abhat and Malatidis [36]
	$CaCl_2 \cdot 6H_2O$	Perkin Elmer DSC 2	Abhat and Malatidis [36]
101-500	Paraffin wax 53	Electric hot plate setup	Sharma et al. [26]
			Sharma et al. [27]
	Acetamide	Electric hot plate setup	Sharma et al. [26]
			Sharma et al. [27]
	Acetanilide	Shimadzu DSC-60	El-Sebaii et al. [42]
	Myristic acid	2 thermostatic bath setup	Hasan and Sayigh [37]
	Stearic acid	2 thermostatic bath setup	Hasan and Sayigh [37]
		Electric hot plate setup	Sharma et al. [26]

			Sharma et al. [27]
	Glauber’s salt	Thermal cycling chamber	Marks [59]
	$MgCl_2 \cdot 6H_2O$	Electric Oven	El-Sebaii et al. [42]
	$NaCH_3COO \cdot 3H_2O$	Thermal bath	Wada et al. [61]
	Capric acid (65 mol%) + lauric acid (35 mol%)	Thermostatic water bath setup	Dimaano and Escoto [50]
		Electric hot plate setup	Shilei et al. [53]
	Caprylic acid (70 wt%) + 1-dodecanol (30 wt%)	Thermostatic chamber setup	Zuo et al. [56]
	Lauric acid (75.5 wt%) + stearic acid (24.5 wt%)	Thermostatic chamber setup	Sari et al. [51]
	Myristic acid (58 wt%) + palmitic acid (42 wt%)	Thermostatic chamber setup	Sari et al. [51]
	Palmitic acid (64.2 wt%) + stearic acid (35.8 wt%)	Thermostatic chamber setup	Sari et al. [51]
501-1000	Paraffin ($C_{22}H_{44.1}$)	Thermostatic bath setup	Hadjieva et al. [28]
	Paraffin ($C_{23}H_{48.4}$)	Thermostatic bath setup	Hadjieva et al. [28]
	Paraffin wax 58-60	Oven	Shukla et al. [29]
	Paraffin wax 60-62	Oven	Shukla et al. [29]
	Erythritol	Oven	Shukla et al. [29]
	Lauric acid	Thermostatic chamber setup	Sari and Kaygusuz [40]
	Palmitic acid	Thermostatic chamber setup	Sari and Kaygusuz [40]
	Stearic acid	Thermostatic chamber setup	Sari and Kaygusuz [40]
	$CaCl_2 \cdot 6H_2O$	Two tube water circulation setup	Kimura and Kai [60]
		Water bath setup	Fellchenfeld et al. [44]
		TA Instruments DSC Q-100	Tyagi et al. [48]
	Al-34%Mg-6%Zn alloy	Thermostatic chamber setup	Sun et al. [57]
	Myristic acid + glicerol	Thermostatic chamber setup	Sari et al. [55]

	Palmitic acid + glicerol	Thermostatic chamber setup	Sari et al. [55]
	Stearic acid + glicerol	Thermostatic chamber setup	Sari et al. [55]
	$CaCl_2 \cdot 6H_2O$ compounds	Perkin Elmer DSC 2	Kimura and Kai [46]
	$Mg(NO_3)_2 \cdot 6H_2O$ (93 wt%) + $MgCl_2 \cdot 6H_2O$ (7 wt%)	DSC	Nagano et al. [47]
	Microencapsulated paraffin wax 53 (0.1g)/polyaniline (0.9g)	Thermal box with heater and cooler setup	Silakhori et al. [62]
	Microencapsulated paraffin wax 53 (0.2g)/polyaniline (0.8g)	Thermal box with heater and cooler setup	Silakhori et al. [62]
	Capric acid (62 wt%) + tetradecanol (38wt%)	Two thermostatic bath setup	Jingyu et al. [63]
	Tetradodecanol (53.60 wt%) + lauric acid (46.40 wt%)	Two thermostatic bath setup	Jingyu et al. [63]
	Tetradodecanol (71.84 wt%) + myristic acid (28.16 wt%)	Two thermostatic bath setup	Jingyu et al. [63]
1001-3000	Paraffin wax 53	Electric hot plate setup	Sharma et al. [27]
	Acetamide	Electric hot plate setup	Sharma et al. [27]
	Myristic acid,	Thermostatic bath setup	Sari [33]
	Lauric acid	Thermostatic bath setup	Sari [33]
	Palmitic acid	Thermostatic bath setup	Sari [33]
	Stearic acid	Thermostatic bath setup	Sari [33]
		Electric hot plate setup	Sharma et al. [27]
	$MgCl_2 \cdot 6H_2O$	Heraeus D-6450 Electric Oven	El-Sebaii et al. [42]
	(NH_4NO_3)(85%) + $(NH_4Al(SO_4)_2 \cdot 12H_2O)$(15%)	Calorimeter + thermal bath setup	Jotshi et al. [49]
	Lauric acid (66 wt%) + myristic acid (34 wt%)	Thermostatic chamber setup	Sari [52]
	Lauric acid (69 wt%) + palmitic acid (31 wt%)	Thermostatic chamber setup	Sari [52]
	Myristic acid (64 wt%) + stearic acid (36 wt%)	Thermostatic chamber setup	Sari [52]
	Paraffin (70 wt%) + Polypropylene (30 wt%)	Electric hot plate setup	Alkan et al. [30]
	Palmitic acid (80 wt%) + expanded graphite (20 wt%)	Thermal cycler BIOER TC-25/H model	Sari and Karaipekli [41]

3001-6000	$C_{17}H_{36}$	Electric hot plate setup	Sari et al. [31]
	Capric acid (55 wt%) + expanded perlite (45 wt%)	Electric hot plate setup	Sari and Karaipekli [34]
	$CaCl_2\cdot6H_2O$	Thermostatic chamber	Porosini [45]
	Glauber’s salt (Na2SO4·$10H_2O$)	Thermostatic chamber	Porosini [45]
	$Na_2SO_4\cdot1/2NaCl\cdot10H_2O$	Thermostatic chamber	Porosini [45]
	$NaOH\cdot3{,}5H_2O$	Thermostatic chamber	Porosini [45]
	Capric acid (73.5 wt%) + myristic acid (26.5 wt%)	Two water baths setup	Karaipekli et al. [54]
	Capric acid + (83 wt%) + stearic acid (17 wt%)	Electric hot plate setup	Karaipekli et al. [35]