

Review

An Overview of the State of the Art and Challenges in the Use of Gelling and Thickening Agents to Create Stable Thermal Energy Storage Materials

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Citation: Prabakaran, R.; Dhamodharan, P.; Sathishkumar, A.; Gullo, P.; Vikram, M.P.; Pandiaraj, S.; Alodhayb, A.; Khouqeer, G.A.; Kim, S.-C. An Overview of the State of the Art and Challenges in the Use of Gelling and Thickening Agents to Create Stable Thermal Energy Storage Materials. *Energies* **2023**, *16*, 3306. <https://doi.org/10.3390/en16083306>

Academic Editor: Luisa F. Cabeza

Received: 1 March 2023

Revised: 28 March 2023

Accepted: 6 April 2023

Published: 7 April 2023



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Abstract: Building cooling and heating, solar-powered energy production, energy recovery, and other energy-consuming industries have all seen an increase in the use of cold/hot latent thermal energy storage (LH-TES). Through energy recovery, LH-TES that uses phase-change materials (PCMs) as a storage medium helps to close the energy supply and demand gap and raises the possibility of energy savings. However, the stability, thermal, physical, and chemical properties of the PCM play a major role in how effectively it can be used. In recent years, adding gelling and thickening agents (GTAs) has gained popularity apart from the nanoparticles (NPs) and nucleating triggers (NTs), particularly for the creation of stable PCMs. Therefore, the current work's goal is to provide an overview of how GTAs are used in the process of developing reliable PCMs for TES applications. It has been found that using GTAs not only increased stability but also decreased sedimentation, leakage, and the supercooling degree (SCD). It was noted that the addition of a GTA with a weight percentage of 2–15% resulted in excellent stability with a negligible leakage rate and latent heat reduced by 3.6–35% after only 200 cycles. Furthermore, PCMs for solar-thermal and building heating systems in the medium-temperature range (21–61 °C) were mostly studied for their performance with GTAs, but no study for a cool TES application was reported. Most works have studied inorganic PCM components with GTAs, and a few reports are available for paraffin. However, the GTA blending resulted in reduced thermal performance due to a decrease in thermal conductivity, latent heat, and a rise in viscosity. Further, NTs and NPs with small amounts were seeded into the PCM-GTA for eradicating the SCD with enhanced TC and accelerated energy transfer.

Keywords: thermal energy storage; phase-change materials; stability; gelling and thickening agents; nucleating triggers; nanoparticles

1. Introduction

The growth of thermal energy storage (TES) is increasing day-by-day by offering excellent performance in energy management, which enables it to reduce the energy gap between supply and demand [1]. Among the available TES approaches displayed in Figure 1, latent heat TES (LH-TES) has a higher volumetric energy density and less

weight, aided by phase-change materials (PCMs) as a storage component [2–4]. It is already proven that the addition of LH-TES is beneficial for building cooling, solar-thermal systems, thermal management units (TMUs) in batteries/electronics and transport vehicle cooling, etc. [5–9]. To develop efficient LH-TES, various PCM categories have been utilized, such as inorganic, eutectic, and organic matter, as mentioned in Figure 2 [10,11]. For instance, organic matter includes paraffin wax, fatty acids, and certain alcohols, while inorganics include water, metals, salts, and their hydrates. Eutectic matter is a combination of two substances from the same group or from different groups [12]. Paraffin PCMs originate from the alkane group consisting of straight-chain hydrocarbons. The properties may vary based on the hydrocarbon count with a melting range between 23 °C and 67 °C. Mostly, the paraffin-based PCMs were used in solar heating applications, as they melt in the range of 50–65 °C [13,14].

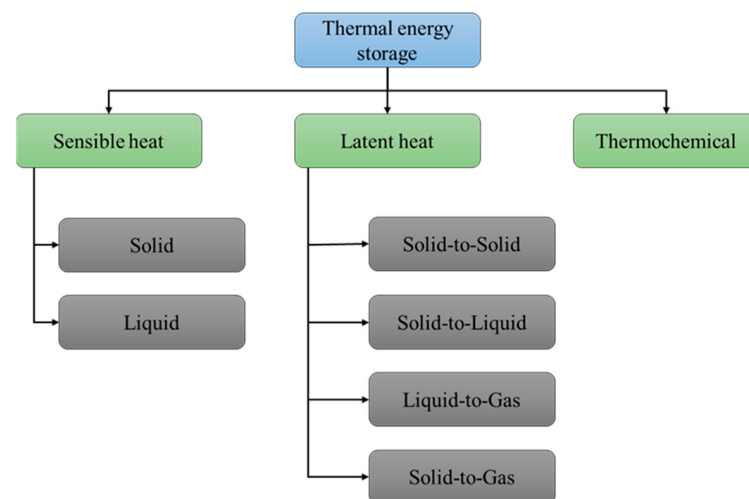


Figure 1. TES technologies with a focus on materials.

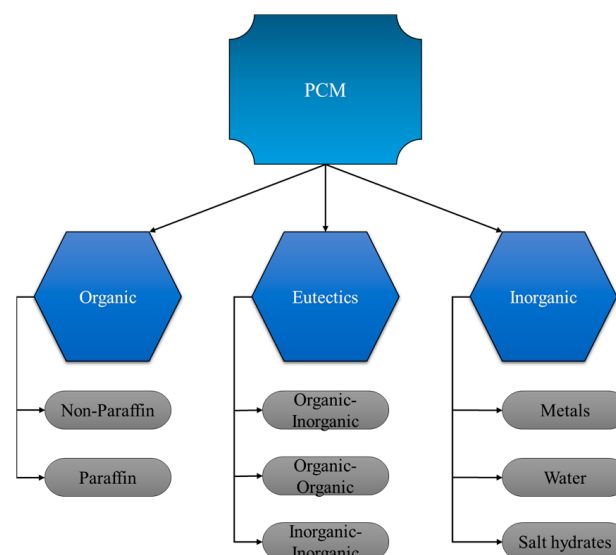


Figure 2. Classification of the PCM.

Compared to organic matter, inorganic has roughly two times higher storage capacity with good numbers in latent heat and thermal conductivity (TC) values and is notably non-flammable in nature [15]. However, these inorganic PCMs were primarily affected by a higher supercooling degree (SCD), which lead to uneven nucleation and further prolonged the phase transition time [16]. Based on the mixing types, eutectics were also found to

have some cons and pros with respect to their own inherent thermophysical properties [17]. Hence, the selection of appropriate PCM involves many critical factors, such as availability, cost, chemical stability, long-term storage cycles, and the hazard factor [18]. Recently, advances in designing LH-TES are encouraged, wherein many techniques have been used to enhance the heat-storing ability of PCM [19]. Despite being a promising PCM type, many complications have been faced during the implementation of PCMs in the LH-TES units, such as leakage, segregation, phase separation, SCD, corrosion, instability (both chemical and physical), lower rate of energy exchange, and lower TC [20,21]. Many technical signs of progress have been discovered to enhance PCM efficiency, namely, nucleating triggers (NTs) (to suppress the SCD), encapsulation (to eradicate the PCM outflow and increase the effective area), fins, and nanomaterials (NMs) (to enhance the thermal properties) [22–26]. Implementing these approaches can help to hasten the energy rate during the heat exchange through the declined SCD and increase the effective area and thermal properties. However, the problems associated with phase separation, volume change, stability, seepage, and shape stability still exist [27].

In the recent years, gelling and thickening agents (GTAs) have attracted some researchers in the PCM field for alleviating the problems related to volume change, seepage, segregation, and shape stability [28]. Gelling is the process of cross-linking PCM with a substance (such as a polymer) to form a three-dimensional network that holds the PCM molecules together, while thickening is the process of incorporating a substance into PCMs to improve viscosity and retain the PCM molecules together [29]. As a result, strong bonding among the molecules will be formed and minimized the leakage during melting. In addition, GTAs also ensure uniform melting during the phase transition process resulting from improved stability despite the higher viscosity of the PCM.

GTAs helps to improve the rigidity and steady blend when mixed with the base materials. Depending on the enhancer, the texture formation was varied from lower to higher viscosities, irrespective of the mixing concentration [30]. Figure 3 illustrates the various applications of the GTAs, including medicinal, food/preservatives, petrochemical, textile, cosmetics, incendiaries and explosives, and TES sectors.

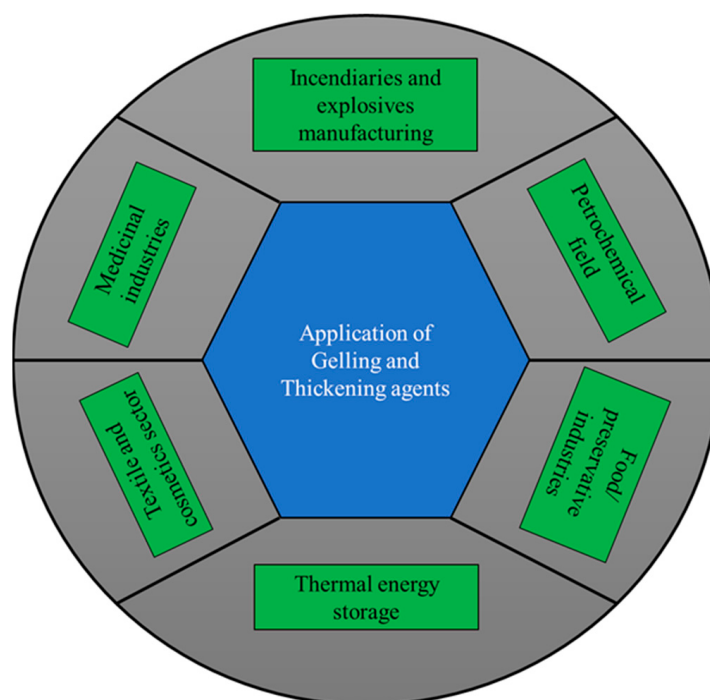


Figure 3. GTA applications in various sectors.

In the medicinal sector, a GTA was employed in the semisolid formulation process to obtain a stable texture (gels) for external drugs [31]. In chemical sectors, the rheological properties of the chemicals are modified by blending them with GTAs, which also helps to improve their stability [32]. The textures of packed food items, namely sauces, salad dressing, soups, liquidity foods, and jams, can be made using GTAs to achieve the desired texture and taste [33]. GTAs (products of guar gum) also help with fluid fracturing in the explosive and petrochemical industries by keeping the proppants inside the fluid [28]. Moreover, GTAs (polyacrylic acid) have been used in textile printing, paints, and colorants [34]. However, the review work conducted by Cong et al. [28] confirms that the usage of GTAs has not been explored to the fullest extent in the LH-TES sector. Therefore, the information on GTA usage in PCM for the LH-TES field are limited, including information on concentration levels, preparation approaches, and underlying mechanisms. As for now, there have been only few comprehensive works reported on the use of GTAs in TES [28], most studies have focused on all the methods employed for the enhancement of the PCM's thermal/physical parameters [35]. Therefore, this review specifically focuses on the use of GTAs in PCMs for LH-TES and reports a thorough assessment covering the following topics, section-wise:

Section 2: GTA classifications based on individual characteristics;

Section 3: GTA mechanisms;

Section 4: Preparation methods for PCM-GTA composites;

Section 5: Application of GTAs in LH-TES—with NTs and with NPs;

Section 6: Summary, challenges, and future scope.

2. GTA Classifications

Based on the material resources, the available GTAs have been categorized into three types, namely (a) natural resource GTAs, (b) synthetic GTAs, and (c) semi-synthetic GTAs; further, these were sub-categorized as (i) organic GTAs and (ii) inorganic GTAs based on their chemical characteristics [32]. The overview of the GTA classifications is illustrated in Figure 4 and the detailed discussions on each category are explained in the succeeding paragraphs.

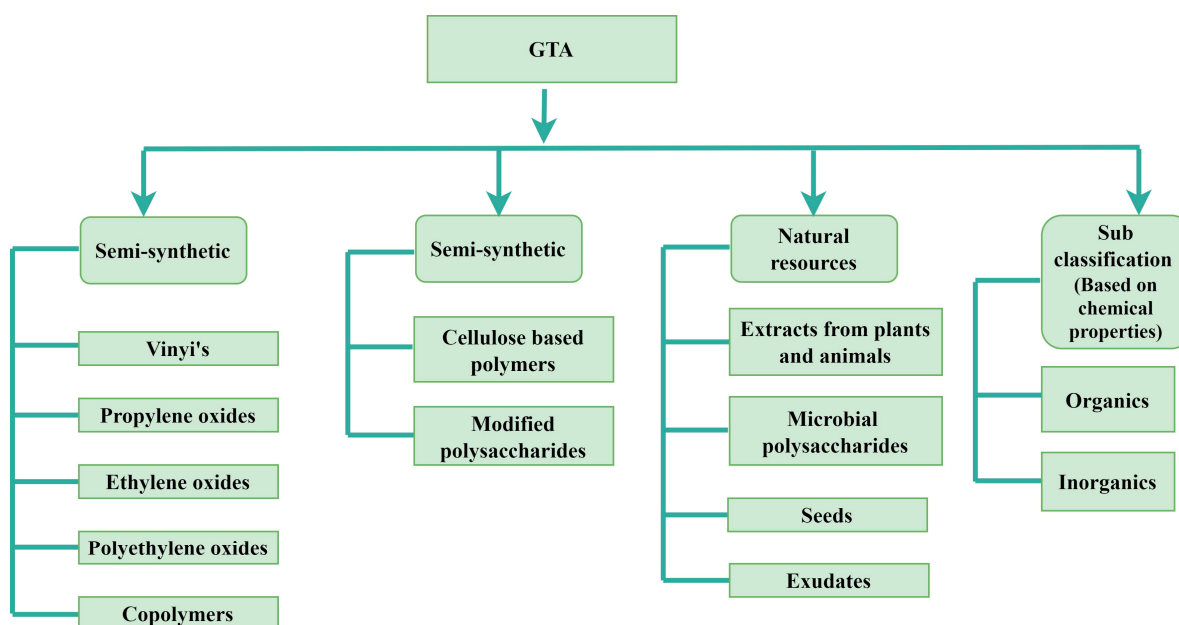


Figure 4. Overview of the GTA classifications.

2.1. Natural GTAs

Natural GTAs have been derived from a variety of naturally plentiful sources, including exudates (from plants and seeds), direct plants, seeds, animal meats, seaweeds, microbial poly-carbohydrates, chitosan, and minerals. Except for minerals, the majority of natural GTAs are made up of organic material. Hydrophilic agar molecules (derived from seaweed) have been used as an addition to increase the capacity of foods to gel while improving stability. However, due to their insoluble nature, these cannot be used in normal or cold water. The hydrating procedure assists in making them soluble in hot water ($>80\text{ }^{\circ}\text{C}$) [36]. Similar to agar, alginates and reversible-nature carrageenan extracted from red and brown seaweeds, respectively, belong to sulfated polysaccharides [37]. Three forms of carrageenans were found, namely, iota, lambda, and kappa, in which iota and kappa are preferred for mild and thick gelling, respectively, while lambda is good for thickening [38]. The kappa form was mostly identified in daily industries (air bubble stabilizing in ice cream and cheese processing), and iota is used for suspending supplementary items in soy milk and salad dressings. Lambda is used in many liquid foods (tomato sauces, syrups, and beverages) for developing texture formation. All forms have good water solubility, while lambda cannot form a gel when mixed with cold water. Lambda is the most expensive of the three forms, and kappa is the most commonly used in food applications [39,40]. Unlike carrageenan, alginates are found to be a versatile GTA, as they can be used for film formatting, stabilizing, and thickening, as well as gelling. Additionally, they have good soluble character in all ranges of water temperature (liquid phase) owing to the existence of calcium ions [38].

Gelatin is mostly processed from animal tissues, and it has a wide range of applications in medicinal and food industries. It has a wider range of structures, from fine particles to flakes, and has superior water-soluble character. It acts as a texturizer or stabilizer, and a thickener in various foods, namely margarine, cheese, and cream [41]. Extracted seed/vegetable products such as guar gum (GG) and xanthan gum (XG) have been identified for thickening purposes, and the latter is the most frequently used. Galactose and mannose are two sugars found in the plant cluster bean and are combined to form GG, while XG is obtained through the fermentation of vegetable leaves of cabbage, cauliflower, broccoli, etc. [42,43]. GG has poor strength in gelling, though it has excellent ability in water thickening compared to other GTAs, and a low wt.% produces more thickening and increases viscosity. A major issue with GG is its unstable nature at lower pH (<3.5) and higher temperatures ($>90\text{ }^{\circ}\text{C}$), and also that it requires polyols and borate ions to reduce the viscosity and augment gelation [28]. The attractive soft texture of XG prompts its usage as a GTA in non-food and food applications, and it is also found as a stabilizer in various solutions (foam, emulsion, etc.) [28]. XGs can easily hydrate in water at normal temperatures, though the dispersion time increases when the temperature and mixing speed fall. Moreover, premixing with water is recommenced for XG before adding the supplementary items (salts) because of its salt tolerance. Among the natural GTAs, XG is mostly employed in LH-TES owing to its superior stability under wide operating conditions (temperature $> 300\text{ }^{\circ}\text{C}$ and $2.5 < \text{pH} < 11$), despite fungal/deprivation issues [44].

2.2. Synthetic GTAs

Synthetic matter is superior to natural GTAs for various applications, such as cosmetics, medicinal and chemical uses, and it provides flexibility in creating specific GTA formulations for wider use. Compared to natural GTAs, synthetic GTAs have longer life, are cheaper, and can produce required quantities with various sizes [32]. Some notable synthetic GTAs are vinyl-based (VB), acyclic-based, and cellulose-based polymers (CB) and copolymers, with CBs being most commonly used in the cosmetics field [32]. VB-based GTAs such as polyvinyl alcohol (PVA) have been found in food-packing applications and have positive aspects, such as being water soluble, having excellent film texturing, and being biodegradable and non-toxic [45]. PVA is used to prevent food spoilage by inhibiting contact between food items and titanium dioxide and oxygen [45,46]. PVA is

easily clingable and stretchable, making it a perfect option for fresh and hot products. However, its poor permeability and moisture-absorbing nature limits its use, and it requires additional material modifications (such as chitosan film, citric acid, etc.) for reducing such issues [47].

A non-ionic derivative polyethylene glycol (PEG) is employed in textiles and medicinal purposes, namely diapers and surgical gauzes [32]. However, there is a possibility of leakage at higher temperatures with PEG in PCMs, but LH modification was insignificant with some PCMs despite an increased viscosity and reduced TC. A wider range of PEG is available based on its molecular weight, with an increase in molecular weight increasing its PCT (phase-change temperature) [48]. Moreover, PEG alone is used as a PCM in various LH-TES applications, despite poor interfacial force and TC. The instability of PEG is prevented by mixing with starch, cellulose, and synthetic GTAs (poly-acrylamide (PAAM) and copolymers) [3].

2.3. Semi-Synthetic GTAs

Cellulose-derivative semi-synthetic GTAs have been identified as esters and ethers with dissimilar properties (mechanical and chemical). These GTAs are known for oxidation, hydrolysis, surface activity, thermo-plastic film nature, and viscosity. Esters are non-soluble in water despite having excellent thickening, while ethers have excellent solubility. Some notable ethers (cellulose) are Carboxymethyl cellulose (CMC), hydroxyethyl cellulose, ethyl cellulose, methylcellulose, and sodium CMC, while cellulose acetate butyrate, hydroxypropyl methylcellulose phthalate, cellulose acetate phthalate, and cellulose acetate belong to esters [32]. The chemical/mechanical properties of the natural starches have been improved by esterification, cross-linking, and grafting, in which the water resistance of a natural GTA (starch) was altered through esterification by using epoxides. The starches' adhesion nature (utility), tensile strength, and water/heat resistant stability were improved via cross-linking (forming a covalent bond (non-polar) among hydroxyl starches) [49]. PAAM is another notable GTA and is mostly used in paper manufacturing (for strengthening), electrophoresis (as a biomaterial), water purification (as a flocculent), medicine (as a hydrogel), and oil recovery fields (as an enhancer) [50,51]. Some studies have been found with PAAM as a GTA for PCMs through developing hybrid PCM aerogels [52]. However, the major issue with semi-synthetic GTAs is their tendency to be prone to microbial contamination, and it is also suggested to hydrate them before use with PCMs; these were mostly used for making lotions, shampoos, and creams [53]. Among the various semi-synthetics, CMC is mostly employed for many purposes as an emulsifier, GTA, and stabilizer. However, it is recommended for cold LH-TES because of the weak gel formation at higher temperatures. Some notable properties and applications of the available GTAs are listed in Table 1. It is noted that most of the GTAs have good water solubility and are reversible. Further, some of them were not recommended for acidic solutions (e.g., agar and gelatin), and some others were not good for high-temperature applications (e.g., PEG, GG, and XG). The properties such as density and viscosity of each GTA can be varied based on their derivatives, hence the general properties for their workability and application were only mentioned. Moreover, no specific approach was reported on the selection criteria for the PCM-TES application, and this should be explored in the future. Therefore, the effect of gelling mechanisms followed by sample preparation and a start-of-art review on the application of GTAs in PCM-TES were described in detail.

Table 1. General properties of available GTAs from the existing studies [28–53].

GTA Type	Structure	Ionic Charge	Solubility	Limitation	Working Nature	Application (Industries)
Agar	Linear/hetero-polymers	Non-ionic	Only in hot water	Not recommended for acidic solutions (pH < 4)	Reversible	Food, medical, and leather
Alginates	Linear/hetero-/homo-polymers	Anionic	Water (except lambda in cold water)	Salt solution (base) might cause the gelling formation	Reversible	Food and medical
Gelatin	Protein-based biopolymer	Covalent compounds	Water soluble	Poor mechanical properties and short degradation times	Reversible	Food and medical
GG	Hydrophilic biopolymer	Non-ionic	Water soluble	Unstable nature at lower pH (<3.5) and higher temperatures (>90 °C)	Reversible	Food, cosmetics, paper, and medical
XG	Hetero-polymers	Anionic	Water soluble	Not recommended in heat-produced (>300 °C) process and (high) salt solvents	Shear-thinning/reversible pseudo-plasticity	Food
PVA	Linear aliphatic	Cationic	Water soluble	Poor permeability and moisture-absorbing nature	Shear-thinning/reversible	Food
PEG	Linear (semicrystalline polyether)	Non-ionic	Water soluble	May cause leakage at higher temperatures, poor loading capacity	Shear-thinning/reversible	Textile and medical
PAAM	Linear-chain or cross-linked	Cationic	Water soluble	Prone to microbial contamination and more difficult to prepare and handle than other GTAs	Shear-thinning/reversible	Medical, oil recovery, and waste-water treatment
CMC	Linear	Anionic	Water soluble	Poor antimicrobial properties	Shear-thinning/reversible	Medical

3. GTA Mechanism

Gelling and thickening processes are almost interwoven, in which the former increases the viscosity of the PCM and the latter helps to hold PCM molecules through interlinking. However, the mechanisms that prevail in both processes are interconnected, and sometimes it is hard to differentiate them because they share similar processes and the level of interlinked force can vary [28]. The level of interlinking is low in thickening, while it peaks during gelling, and the mechanism that prevails during the viscosity increments can vary depending upon the GTA used. Apart from the GTA's nature, external (environment) factors, namely GTA concentration/molar mass, pH level, ionic strength, pressure,

enzyme existence, solvent quality, and temperature, can also affect gel and thickening formation [54,55]. Overall, three major mechanisms have been reported during the mixing of GTAs and the base fluid [28], as shown in Figure 5. Interlacing and physical mechanisms are involved in thickening, while physical and chemical mechanisms are involved in gelling.

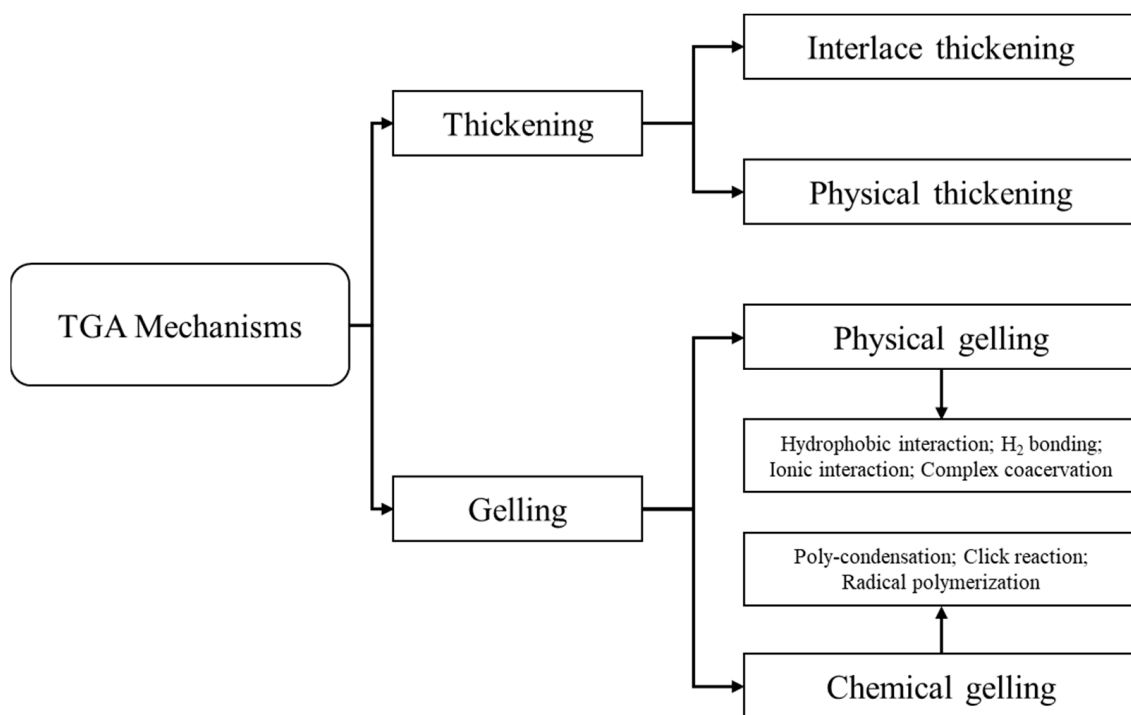


Figure 5. GTA mechanisms [28].

- **Interlace thickening:** This mechanism is observed in natural GTAs and is characterized by a high elastic nature with pseudo-plastic behavior. The increase in viscosity during thickening occurs due to the entangling of heavy polymer molecules in the solution. The efficacy of the thickening process is determined by the molecular weight of the polymer [56].
- **Physical thickening:** This mechanism is observed in good water-soluble GTAs and light-molecular-weight molecules, which make an interconnection with one another to form a texture, resulting in an increase in viscosity [57]. Additionally, the interconnection strength depends on the GTA nature: if the strength is higher it leads to an increase in viscosity at a higher rate.
- **Physical gelling:** This mechanism is experienced in gel with reversible nature, and external conditions such as temperature, pressure, and pH significantly affect the physical gelling [58]. H₂ bonding can be achieved by modulating the temperature or pH of the water-based solution. The repulsion effect between hydrophilic and hydrophobic components results in hydrophobic gelling. Similarly, the interfacing of anionic and ionic components leads to coacervation gelling.
- **Chemical gelling:** This mechanism occurs due to the reaction among the functional components, creating covalent chemical gelling. The type of chemical gelling varies based on the GTA's nature and corresponding functionalization [58]. Redox reactions due to the formation of free radicals produce radical polymerization gelling, while click reaction gelling is mostly employed in biological fields because of its efficiency, strong gelling, and hastened reaction kinetics even at medium temperatures.

4. PCM + GTA Preparation

The preparation of PCM + GTA can be performed using three approaches: (i) liquid PCM (melt) mixing (LPM), (ii) solution (solid PCM) mixing (SM), and (iii) dry PCM mixing (DPM). The selection of the preparation method among the three approaches mostly depends on the usage of the base PCM and its composites. More importantly, the concentration of each constituent should be properly measured before preparation [28]. The layout of each preparation approach is displayed in Figure 6. In the LPM method, first the selected PCM needs to be in pure liquid form and heated at a constant temperature ($>PCT$ of the PCM). Then, the required mass of the GTA in powder form is added to the PCM liquids and allowed to undergo the stirring process until proper mixing is achieved. The time taken for the stirring process varies for each type of PCM and GTA used.

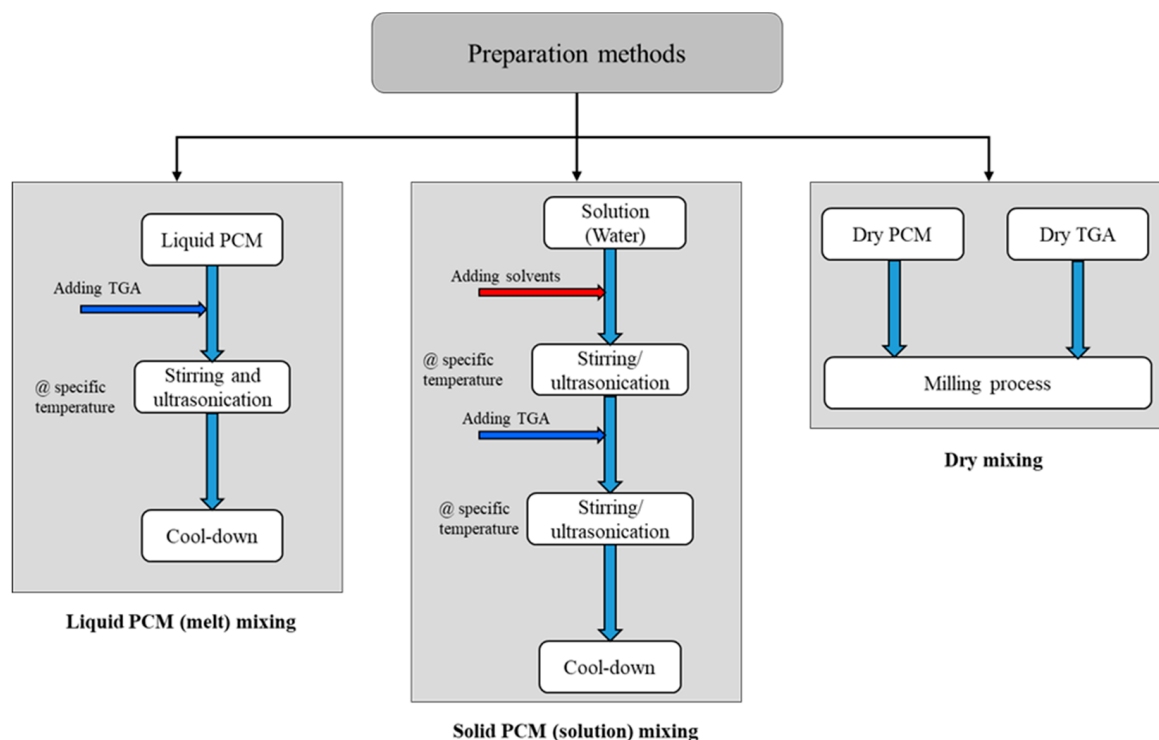


Figure 6. PCM + GTA preparation approaches [28].

The SM method is usually applied for solid PCM (example: salt hydrates) + solution mixtures (water). First, the required quantity of the solid PCM is added into the liquid water to undergo stirring to ensure homogenous mixing, followed by a GTA seeded into the PCM solution. In the DPM method, both the PCM and GTA are dry powder, and both powders (usually different sized) are mixed using the milling process to aid uniform mixing. Mostly, the LPM and DPM approaches are employed owing to the simplified preparation steps. For instance, Saeed et al. [59] blended FA and Methyl Palmitate (MP) (60/40 by molar ratio) at 50 °C, followed by 10% (by mass) of 2-hydroxypropyl ether cellulose (HPEC) addition, and the sample was stirred for 30 min at the same temperature. After ensuring proper mixing, the sample was softened through heating at 70 °C, and then 10% (by mass) of graphene nanoplatelets (GNPs) was added into PCM/HPEC sample to enhance the TES performance. However, Cong et al. [28] mention that if the PCM is planned to make with NTs and NPs to improve TC and reduce the SCD, it is recommended to add the GTA at the end. Similarly, Wang et al. [60] mixed PAAM into a sodium acetate trihydrate (SAT) + Tetrasodium pyrophosphate decahydrate (TPD) PCM composite (liquid), in which TPD is an NA, while SAT is a PCM.

5. Application of GTA in LH-TES

As mentioned earlier, the addition of a GTA to PCMs can reduce/eliminate phase separation and leakage by forming texture and increasing viscosity. This section discusses some notable observations from previous studies that were reported for the various combinations of PCMs and GTAs.

5.1. PCM + GTA with NTs

In 1980, Marks [61] introduced attapulgite clay (9.3 wt.%) as a GTA in Glauber's salt (GS) + water solution (1:10 ratio) with borax (2.7 wt.%) as an NT. The pure PCM showed poor cyclic stability, with a rapid reduction in LH from 238 to 63 kJ kg⁻¹ after few (40) cycles. This could affect the long-term performance of the LH-TES, but the addition of a GTA showed excellent stability, with 105 kJ kg⁻¹ of LH even after 200 cycles, despite a slight reduction in initial LH. Telkes [62] used the same GS + water + borax-based PCM with inorganic thixotropic material (ITM) as a GTA. The concentration of ITM was varied from 0 to 10% and gel formation was observed with >6% of ITM in the PCM solution; in addition, excellent cyclic (10 cycles) stability was also observed. In another study [63], the CMC (2–4 wt.%) was utilized in an SAT + potassium sulfates (PS)-based PCM to make stable components, and various NTs were tested to diminish the SCD. The observed SCDs of the PCM were 3–4 °C and 0–3 °C with borax (3 wt.%) and PS (2 wt.%), respectively. The same kind of result was also confirmed by Shin et al. [64], and the observed TC, LH, and SCD were 0.64 W m⁻¹ K⁻¹, 227 J g⁻¹, and 3–4 °C, respectively, with borax inclusion (1.9 wt.%). Cabeza et al. [65] employed three GTAs, namely bentonite, cellulose (M and MHE), and starch (wheat flour), in an SAT-PCM. A drastic drop in LH was observed (20–35%) despite a similar PCT, and MHE-cellulose showed good stability until 65 °C as compared to other GTAs.

The improper melting (stability) of disodium hydrogen phosphate dodecahydrate (DHPD) was enhanced by adding 3–5 wt.% of sodium alginate (thickener) or polyacrylate sodium (gelling) [66]. The results showed that the use of sodium alginate eliminated phase separation, while polyacrylate sodium seeding prevented phase degradation. Both amylose and sodium alginate grafted sodium acrylates showed excellent stability after a certain number of thermal cycles [67,68]. CMC, as a GTA, was used in the salt hydrate mixture containing 60% magnesium nitrate hexahydrate (MNH) and 40% magnesium chloride hexahydrate (MCH) by Qing et al. [69] and examined for its impact by varying the wt.% from 1 to 20. Notably, good stability was observed with 5 wt.% of CMC, despite a 32% reduction in storage capacity. However, El-Sebaai et al. [70] employed additional water with a sealed container for an MCH-PCM, which helped to avoid the reduction in LH due to the GTA addition. Combining one kind of GTA (CMC) with another (silica gel) was also carried out by Ramirez et al. [71] to enhance the SAT stability. In a GTA concentration (<3 wt.%), 15% of silica gel was mixed with 85% of CMC. The results showed an increment in melting cycle with a reduction in CMC wt.%, while the SCD decremented notably. Further, the chemical structure of CMC helped prevent segregation during phase change with reduced crystallization energy. However, the increased silica gel proportion eased fusion but not crystallization. Finally, the combination of two GTAs aided in enhancing LH recovery.

Ushak et al. [72] studied the performance of a PCM (inorganic salt)-based TMU for batteries by adding PEG 600 (5 wt.%) with various NTs. Among the salts, GS was found to be the best PCM option owing to its satisfactory stability after six cycles. PEG 600 (5 wt.%) reduced the LH of the PCM from 220.5 to 181.6 J g⁻¹, and the PCT marginally rose from 36.8 to 37.7 °C. Further, 5 wt.% of boric acid was added as an NT to reduce the SCD, resulting in a 5.6% increase in LH. Alkan et al. [73] elucidated the significance of PVA in the TES performance of calcium chloride hexahydrate (CaCl₂·6H₂) (CCH) and GS. The DSC of freezing, and melting of the CCH and GS with PVA are illustrated in Figure 7. According to Figure 7a,b, the LH observed during energy storage (freezing) and the release (melting) of GS + PVA is greater than CCH + PVA by 67.8% and 71.1%, respectively. This could be due to the superior water-soluble nature of GS compared to CCH. As shown in Figure 7c,d,

the change in temperature for CCH + PVA is always faster than GS + PVA, which could be due to the higher TC and lower LH of the CCH.

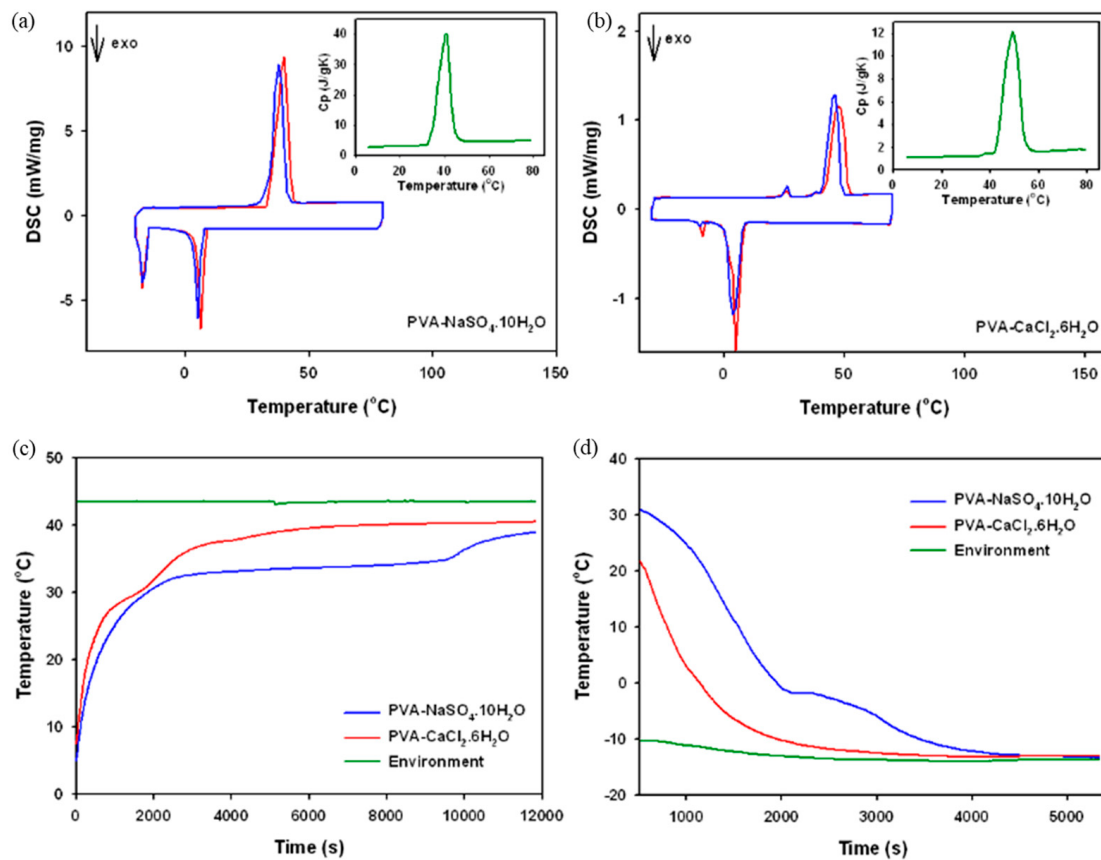


Figure 7. (a) DSC of GS + PVA; (b) DSC of CCH + PVA; (c) freezing and (d) melting of GS and CCH with PVA [73].

Karimineghlani et al. [74] compared the performance of lithium nitrate trihydrate (LNH) with PVA (15 wt.%) and compared its gel formation with a water solution + PVA (15 wt.%). The visual analysis regarding the gel formation between LNH + PVA and water + PVA is illustrated in Figure 8a, which showed an easy flowing nature of water + PVA, while mild gel formation was seen with LNH + PVA without crystals. The gel formation of LNH + PVA as a function of temperature during the cooling/heating cycle (shown in Figure 8b) indicated the solid-to-gel transition occurrence at ~20 °C. Further, the leakage result of LNH with and without PVA is displayed in Figure 8c. It is observed that the seeding of PVA had an excellent result in holding the LNH molecules together and restricting the leakage. Eutectic PCM strengthening (stability) was conducted by including sodium alginate (0–5 wt.%) [75]. The DSC results showed a reduction in LH by 35.1% with 5 wt.% of sodium alginate, and freezing onset was also prolonged by 2.9 °C. More importantly, incremented viscosity with GTA addition causes the PCM diffusion and minimizes the crystallization energy, leading to a reduced SCD. The significance of five NTs and gelatin (as a GTA) on a BHO-PCM was examined by Wang et al. [76] for a hot LH-TES application. Adding calcium fluoride (1 wt.%) provided superior performance among the various NTs with gelatin (1 wt.%), with a lower SCD (1.8 °C) and excellent LH (343 kJ kg^{−1}) and TC (0.96 W m^{−1} K^{−1}).

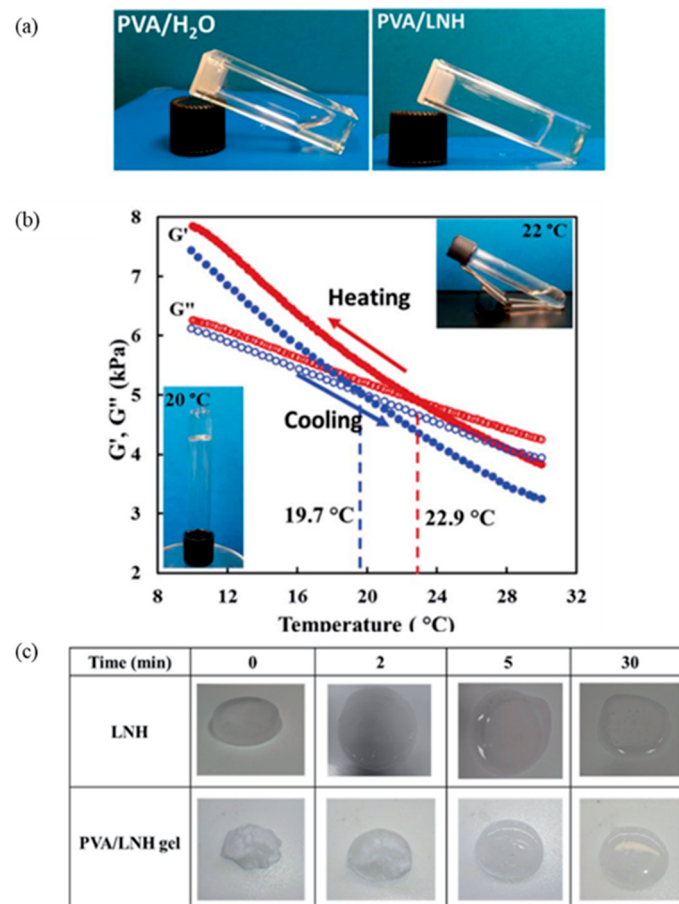


Figure 8. (a) Photographic view of water + PVA and LNH + PVA samples; (b) heating and cooling curves; (c) melting of LNH with and without PVA [74].

In order to attain a stable and reliable thermal performance of DHPD, Peng et al. [77] utilized silica (fumed) as a GTA, and the photographic views of DHPD samples with 2 wt.% of silica (fumed) are illustrated in Figure 9. The effect of FS inclusion on the melting, freezing, and TC of the DHPD is displayed in Figure 10a–c, respectively. It is perceived that the PCTs of the pure and GTA-added DHPD were found to be 35.5 °C and 33.6–35.2 °C, respectively. More importantly, a steady phase was only seen with pure and 2 wt.% seeded DHPDs, which ensured that 2 wt.% addition of a GTA (silica) was better than other loadings. Despite a reduced SCD from 14.4 to 8.7 °C (Figure 10b), there were no changes in the total melting period (40 min), while the solidification was prolonged. This could be due to the increased velocity, which limited the freezing progress despite increased TC (Figure 10c).

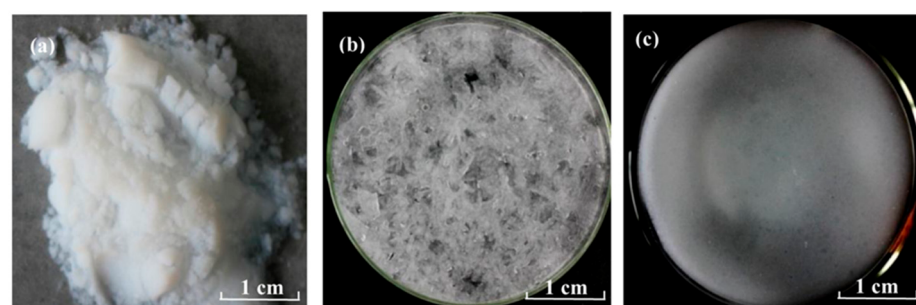


Figure 9. (a) Silica (fumed); (b) DHPD; and (c) DHPD + silica (2 wt.%) [77].

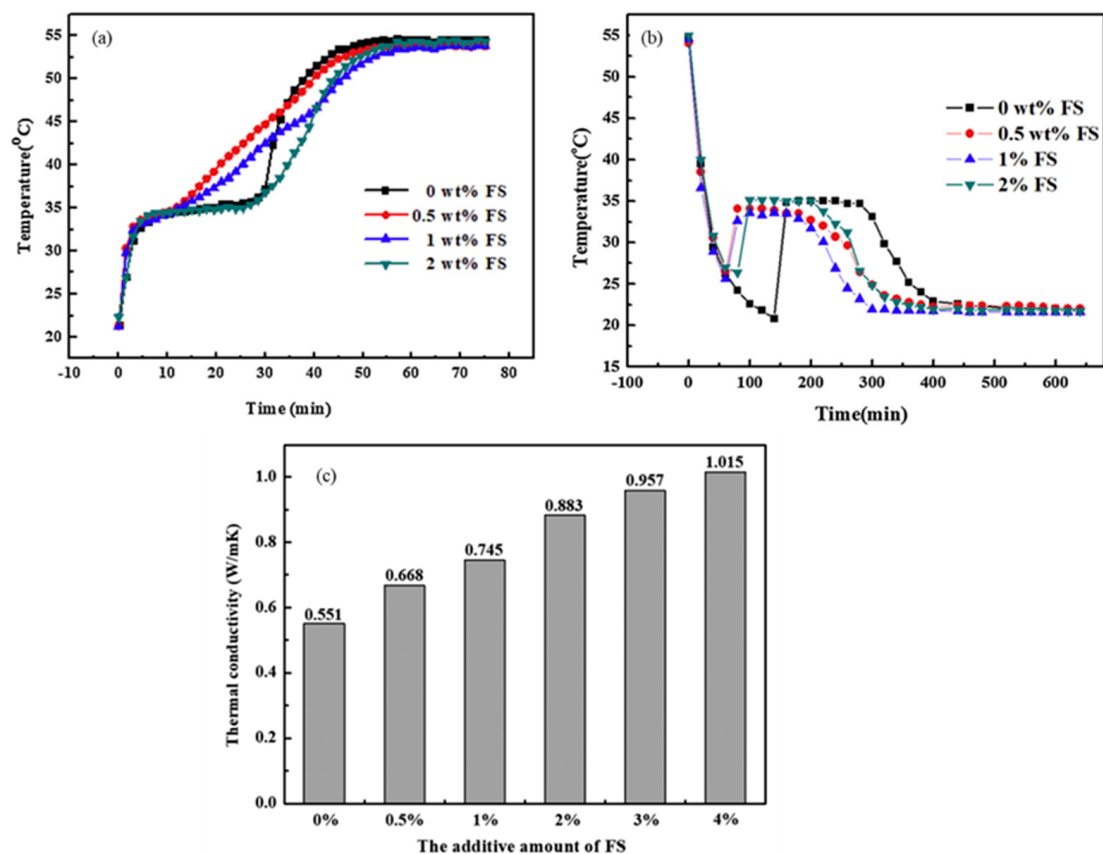


Figure 10. (a) Melting cycle; (b) freezing cycle; and (c) TC of the DHPD samples [77].

Important observations from existing PCM + GTA + NA studies are consolidated in Table 2. Overall, the inclusion of a GTA alone helped prevent seepage and provided better stability for the PCMs, with some even reducing the SCD notably. Additionally, the NT seeding along with a GTA in the PCM eliminated the SCD and ensured crystallization with reduced energy. However, there was a reduction in LH and TC with increased viscosity, which limited the loading level of the GTA. Additionally, it does not hasten the melting/solidification process. Therefore, further studies need to be conducted to address these issues through the addition of NPs along with a GTA into the PCMs.

Table 2. Notable observations from the existing PCM + NT+ GTA studies.

PCM	GTA/wt. %	NT/wt. %	Preparation Approach	Observations				Application	Ref.
				PCT Melting (°C)	SCD (°C)	TC (W m ⁻¹ K ⁻¹)	LH (kJ kg ⁻¹) (No. of Cycles)		
GS	Attapulgate clay/9.3	Borax/2.7	SM	32.4	7	-	105 (200)	Space heating	[61]
SAT	CMC/3	PS/2	LPM	58	0–3	0.6	206 (200)	Solar heating	[63]
	SAP/3	Borax/3		32	3–4	0.64	227 (200)		
DHPD	Sodium alginate	-	SM	35.4	-	-	260.9 (-)	-	[68]

Table 2. Cont.

PCM	GTA/wt. %	NT/wt. %	Preparation Approach	Observations				Application	Ref.
				PCT Melting (°C)	SCD (°C)	TC ($W m^{-1} K^{-1}$)	LH ($kJ kg^{-1}$) (No. of Cycles)		
MNH + MCH (60:40)	CMC/5	-	LPM	57	-	-	100 (100)	Solar heating and building	[69]
SAT	CMC + silica gel (85:15)/3	Silver	-	62.13	-	-	285.8 (10)	Solar heating	[71]
GS	PEG/5	Boric acid/5	-	34.9	-	-	191.8 (-)	Battery	[72]
CCH GS	PVA	-	-	42.1 34.2	- -	0.16–0.19	248.7 (-) 80.0 (-)	Green house and food preservation	[73]
SAT	XG/0.5 CMC/1	-	LPM	-	-	0.54 0.64	-	Solar thermal	[78]
SAT	XG/2	Disodium phosphate/-	LPM	58.8	-	-	240.3 (200)	Solar thermal	[79]
Fatty acids	MT-800 (Mil)/3	-	LPM	58.1	-	-	-	-	[80]
GS	Fibrillated cellulose (nano/micro-sized)/1	Borax/5	SM	29	1.9	-	-	-	[81]
Paraffin	Cellulose nanofibers (CNF)/20	-	SM	54.8	9	-	171.4	Building heating	[82]

5.2. PCM + GTA with NPs

With better TC and a more active heat transfer area, NP addition is a cutting-edge method for increasing the energy transfer rate in several thermal utilities, including TES, solar-thermal units, and TMUs [83–87]. Colloidal stability is a negative factor associated with the addition of NPs to the PCM, whereas the addition of a GTA increased the PCM's stability despite lower TC and LH levels. As a result, the research community has also experimented with combining NPs and GTAs to create an effective PCM (enhanced TC with superior stability and tiny SCD), and the results are discussed in this sub-section.

Li et al. [88] prepared an SAT-potassium chloride (PC)-based PCM through the seeding of CMC and aluminum oxide (Al_2O_3) as the GTA and NP, respectively. They varied the wt.% of PC and Al_2O_3 in the range of 0–10 and 0–5, respectively, with 4 wt.% of CMC. The optimum PCM was achieved by feeding the PC, CMC, and Al_2O_3 into the PCM by 8, 4, and 1 wt.%, respectively. The observed SCD, LH, and PCT of the optimum PCM were 0.1 °C, 232.3 J g⁻¹, and 50.45 °C, respectively. Ag (Silver) (0.6 wt.%) and Cu (copper) (0.5 wt.%) NPs were also seeded into SAT with CMC instead of Al_2O_3 , resulting in a similar SCD and LH [71,89]. An efficient GS-PCM was realized by Li et al. [90] through the seeding of CBO, EG (expanded graphite), and CMC. The energy storage/releasing of the GS-PCM with and without EG and CBO is displayed in Figure 11. The feeding of CBO reduced the SCD despite a huge loss in storage level. Further addition of EG helped to regain GS's capacity during storage. Among the various GS-PCM samples, GS + CBO + EG (7 wt.%) produced good results with a 19 °C reduction in the SCD and a 3.6% increment in TC.

A combination of hydrophobic silica (1–5 vol.%) and graphite (1–15 vol.%) was utilized by Gorbacheva et al. [91] for improving the paraffin's TES properties. They observed percolation in the PCM after seeding HS at about 3 vol.%. Further addition of graphite (15 vol.%) aided in good energy performance, with a 33% of TC augmentation. Dong et al. [92] used a PEG-PCM with copper sulfide and PVP (polyethylene pyrrolidone) for photo-thermal utilization, and melt mixing was employed for sample preparation. They varied the concentration of copper sulfide from 2–10 wt.% with 2 wt.% of PVP in PEG. They observed a reduction in LH from 171.8 to 156.2 J g^{−1}, while TC incremented from 0.235 to 0.247 W m^{−1} K^{−1}.

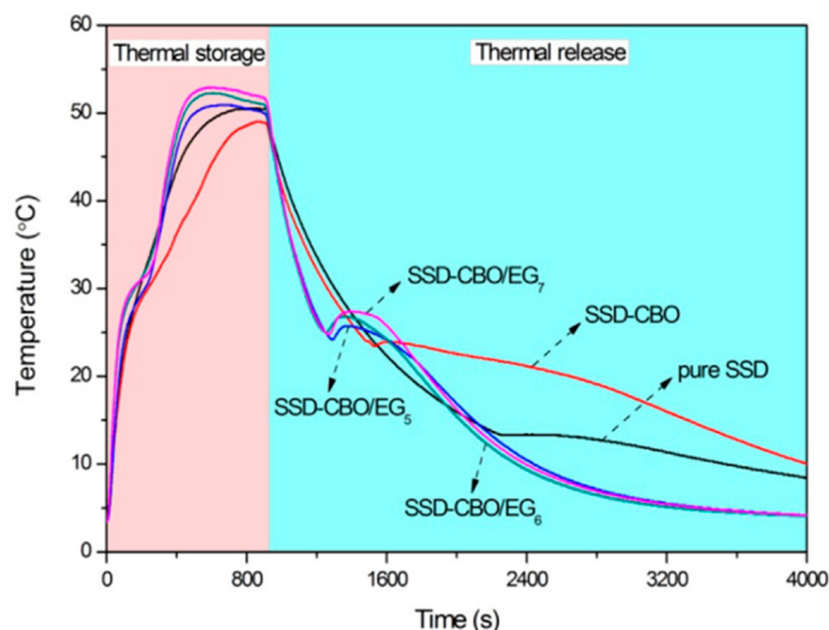


Figure 11. Energy storage/release of the GS-PCM with/without CBO and EG [90].

Carbon NPs (CNPs) were found to be superior to metal NPs (MNPs) [93,94], providing an excellent increase in TC with minor modifications in the density and viscosity owing to their excellent active heat transfer area. It has also been shown that CNPs lead to higher TC increments compared with MNPs [95–98]. Liu et al. [99] prepared a new PCM comprising GS (PCM), PAAS (GTA), and MWCNTs (multi-walled carbon nanotubes), and mechanical mixing was employed during sample production, as shown in Figure 12. Their results showed a 141.7% enhancement in TC due to the MWCNT seeding (3 wt.%) into the GS-PAAS PCM, and only 3.6% of LH was reduced even after 500 thermal cycles. In another study, 1–10 wt.% of GNPs was added along with HPEC (10 wt.%) in a PCM mixture (fatty acids +MP) [59]. As a result, TC was increased by 102% with GNPs (10 wt.%) despite a 7% LH drop; more importantly, there was only a minor change in the PCT with a 0.1 °C SCD. Furthermore, the PCM passed the thermal reliability test, showing consistent thermal performance over 30,000 thermal cycles with no appreciable variations in the PCT and LH.

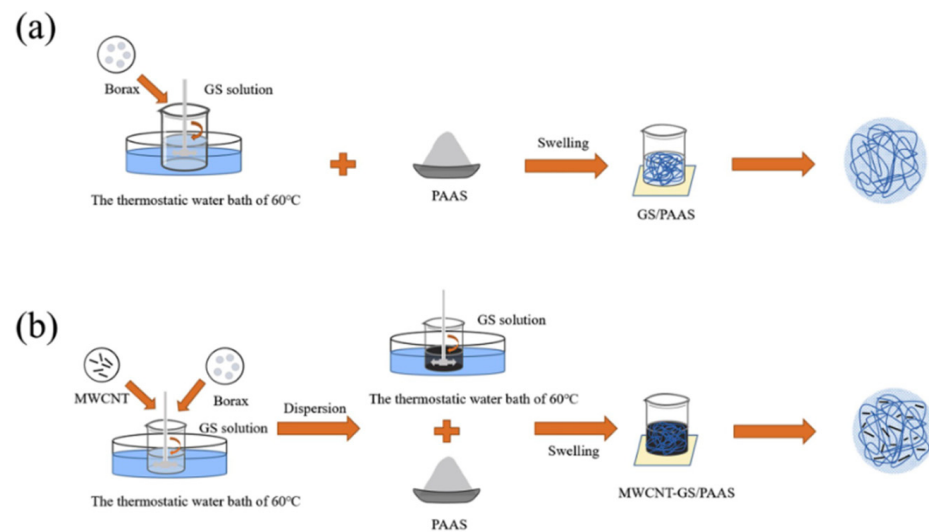


Figure 12. Sample preparation approach used by Liu et al. [99]: (a) PCM + GTA and (b) PCM + NM + NA + GTA.

Wei et al. [100] developed a hybrid structure using GNPs and CNFs in a PEG-PCM for TMUs. The solution mixing approach followed by sonication and vacuum drying were used for preparing the PEG-GNP-CNF samples, as shown in Figure 13. The results of the PEG + CNF-GNP samples, namely heating, cooling, melting LH, and cyclic stability, are displayed in Figure 14. The analysis revealed only marginal changes in the peak point during melting/cooling, and a 12.6% drop in LH was observed with 4 wt.% GNPs. However, the presence of network formation resulted in great cyclic stability over 500 cycles.

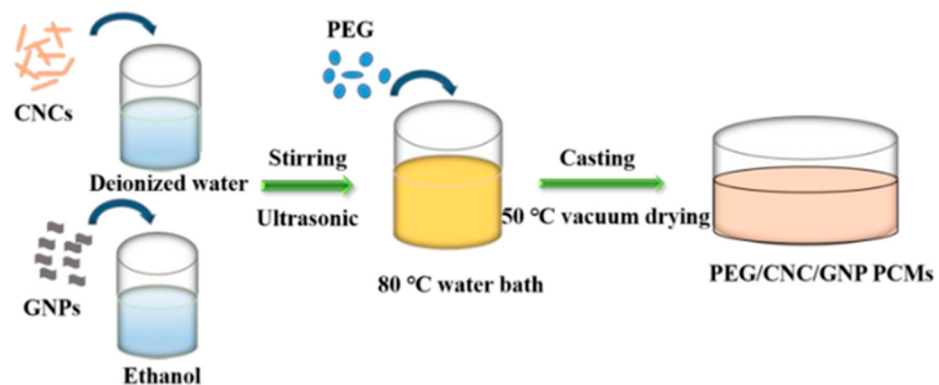


Figure 13. Sample preparation approach followed by Wei et al. [100].

Vasilyev et al. [101] used a hybrid GTA containing MIL and HAS (in a ratio of 17:83 by mass) in a bio-PCM to increase its stability. Additionally, GNPs and MWCNTs were added separately as NPs to enhance the TC of the bio-PCM + GTA. Initially, the GTA concentration was varied from 0 to 15 wt.%, and it was observed that leakage of the samples was negligible (2%) with 15 wt.% of the GTA, as shown in Figure 15a. Further, leakage of the PCM-GTA samples with MWCNTs and GNPs was also examined (Figure 15b), and it was observed that the sample with the GTA and GNPs showed a very low leakage (2%), while the sample with MWCNTs had a notable leakage. The heating and cooling results from Figure 15c,d, respectively, showed that the GNP-containing PCM sample hastened the process owing to the excellent TC augmentation compared to the other samples. The overall observations from the PCM + GTA with NPs are listed in Table 3. It was inferred that the drawbacks of reduced TC and slower energy rates during storage/release due to the GTA addition were

significantly eliminated with the seeding of NPs into the PCM + GTA samples; furthermore, the NPs also acted as NTs and suppressed the SCD.

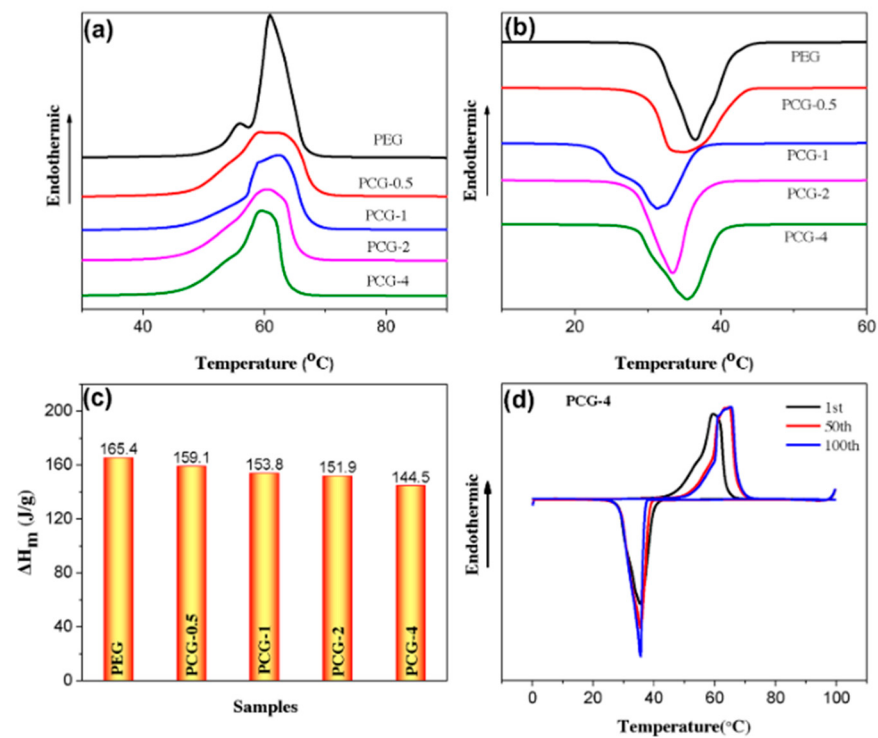


Figure 14. (a) Heating DSC; (b) cooling DSC; (c) melting LH; and (d) cyclic analysis [100].

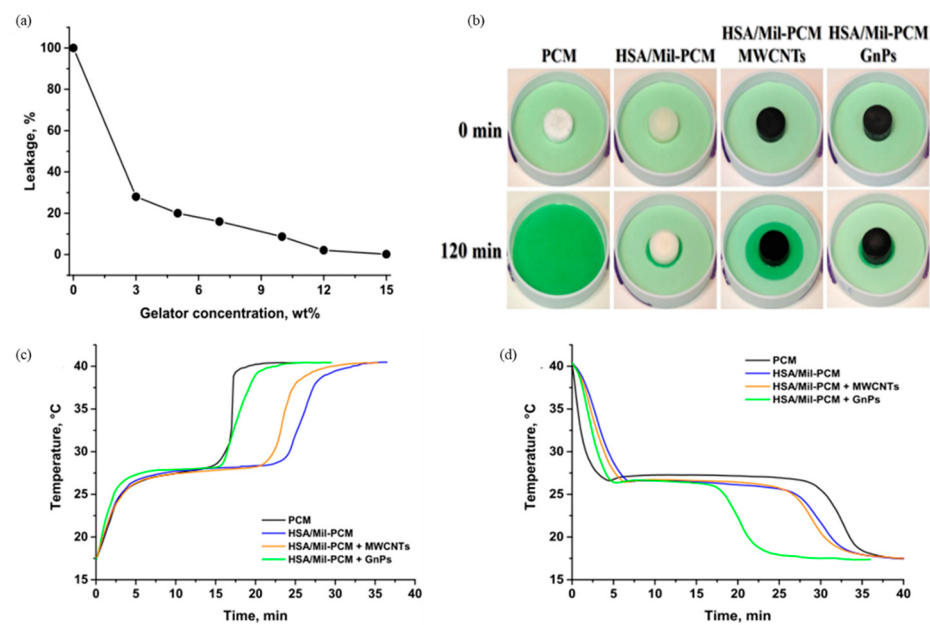


Figure 15. (a) GTA versus leakage rate; (b) leakage of the samples used; (c) heating curve; and (d) cooling curve [101].

Table 3. Notable observations from the existing PCM + NT+ GTA studies with NPs.

PCM	GTA/wt. %	NP/wt. %	Preparation Approach	Observations				Application	Ref.
				PCT Melting (°C)	SCD (°C)	TC (W m ⁻¹ K ⁻¹)	LH (kJ kg ⁻¹) (No. of Cycles)		
SAT + PC	CMC/4	Al ₂ O ₃ /1	Simple blending	50.45	0.1	-	232.3 (50)	Building and water heating	[88]
SAT	CMC/3	Cu/0.5	Dispersion in liquid SAT	57.8	0.5	0.936	231.4 (50)	-	[89]
GS	CMC/2	EG/0.7	-	31.06	8.03	1.96	114 (50)	Building envelope	[90]
Paraffin	Silica/3	Graphite/15	LPM	57.5	-	-	131.6	-	[91]
PEG	PVP/2	Cu Sulfide/10		56.23	13.37	0.247	151.8 (100)	Solar thermal	[92]
GS	PAAS/7	MWCNT/3	Mechanical mixing	38.5	-	1.38	180.4 (500)	Solar thermal	[99]
PEG	CNC/8	GNP/4	Physical compound	59.6	-	2.018	145.2 (100)	Electronic devices	[100]
Bio-PCM	Mil + HAS (17:83 by mass)/15	GNP/3 MWCNT/0.6	LPM	26.9–28.3	0.2–0.3	0.454 0.33	-	Green house and water heating	[101]
Mirabilite-PCM	CMC/-	GO + CNF/-	-	-	0.7–1	-	86.3 (1500)	Solar thermal	[102]

6. Summary, Limitations, and Potential Scope of PCMs with GTA

The present work describes a critical assessment of how the stability of the PCMs can be improved with the aid of various GTAs. Prior to discussing the state of art of the PCM-TES performance, a brief discussion about the TES, PCM, and GTA classification was presented. Although the addition of GTAs to the PCMs has been found to improve their physical and chemical stability over multiple cycles, with negligible seepage and a reduced SCD, some limitations concerning viscosity, TC, LH, and energy storage/releasing rates must be addressed before this new type of PCM can be used in real-world applications. Moreover, most of the available GTAs have been seeded with inorganic PCMs, especially salts for hot and medium LH-TES applications, and few have been tried for bio- and organic PCMs (Paraffin). Most of the studies used about 2–15 wt.% of the GTA in the PCM and investigated its stability and leakage rate. Even after 200 cycles, LH reduced by 3.6–35% only with GTA addition, which is significantly better than pure PCMs. Mostly, the PCM + GTA performances have been tested for the application of solar-thermal and building heating systems. Additionally, different types of GTAs require different preparation methods, such as preheating followed by melt mixing, direct mixing, and vacuum drying. Further, numerous studies have used a single GTA, and more lately, hybrid GTAs, which combine two or more types of GTAs, have been increasingly used to improve seepage resistance. To satisfy the expanded TES criteria, more research is needed on the velocity profile, separation of liquid/solid PCM molecules during melting and crystallization, and storage/release rates. In particular, a system-level study considering actual conditions needs to be explored more elaborately to ensure the practicality viability.

Moreover, NTs were seeded with a GTA for the total eradication of the SCD, even though there was still a decreased rate of energy release/storage. In order to accelerate the energy rates during melting and crystallization through enhanced TC, MNPs and CNPs were introduced to the PCM-GTA-NT solutions. However, adding more materials to the PCM still has the potential to increase viscosity, which in turn affects the natural convection. NP sedimentation appears to be the main factor affecting the long-term operation of the TES. Moreover, some studies mixed the NPs early in the preparation process, while others employed them only at the very end; this needs to be addressed in detail. The usage of a water PCM in cold LH-TES for solar-thermal and building cooling systems has

increased recently [103–109], and research on the effects of GTAs, NPs, and NTs on the performance of water PCMs can be exploited to create more energy-efficient components. Furthermore, hybrid NPs may also be useful for PCM-GTA samples to further increase TC. Most of the studies reported the GTA's concentration for achieving the stable PCM. However, external (environment) factors, namely pH level, ionic strength, pressure, enzyme existence, temperature, and solvent quality on the PCM's stability need to be explored. The development of novel PCM-GTAs with various NT and NP types (single/hybrid) for commercial LH-TES applications, together with energy, exergy, and cost-benefit analyses, has tremendous potential. The environmental impact of PCM-GTAs for their entire life cycle using a Life Cycle Assessment for designing cost-effective and sustainable LH-TES systems needs to be explored [110].

Author Contributions: Conceptualization, R.P. and S.-C.K.; methodology, R.P.; data collection and formal analysis, R.P., P.D. and A.S.; writing—original draft preparation, R.P.; validation, writing—review and editing, M.P.V., P.G., S.P., A.A. and G.A.K.; writing—review and editing, S.-C.K.; supervision, S.-C.K. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: Data sharing not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

Nomenclature

CB	Cellulose-based
CCH	Calcium chloride hexahydrate
CNF	Cellulose nanofibrils
CNP	Carbon nanoparticle
DHPD	Disodium hydrogen phosphate dodecahydrate
DPM	Dry PCM mixing
GG	Guar gum
GNP	Graphene nanoplatelets
GS	Glauber's salt
GTA	Gelling and thickening agents
HPEC	Hydroxypropyl ether cellulose
ITM	Inorganic thixotropic material
LH	Latent heat (J g^{-1} or kJ kg^{-1})
LNH	Lithium nitrate trihydrate
LPM	Liquid PCM mixing
MCH	Magnesium chloride hexahydrate
MNH	Magnesium nitrate hexahydrate
MNP	Metal nanoparticle
MWCNT	Multi-walled carbon nanotubes
NP	Nanoparticle
NT	Nucleating trigger
PAAM	Poly-acrylamide
PCM	Phase-change material
PCT	Phase-change temperature ($^{\circ}\text{C}$)
PEG	Polyethylene glycol
PS	Potassium sulfates
PVA	Polyvinyl alcohol
PVP	Polyethylene pyrrolidone
SAT	Sodium acetate trihydrate
SCD	Supercooling degree ($^{\circ}\text{C}$)
SM	Solution mixing
TC	Thermal conductivity ($\text{W m}^{-1} \text{K}^{-1}$)
TES	Thermal energy storage
TMU	Thermal management unit
VB	Vinyl-based
XG	Xanthan gum

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