Composition and Preparation of Microencapsulated Phase Change Materials

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Phase change materials (PCMs) have been extensively utilized in latent thermal energy storage (TES) and thermal management systems to bridge the gap between thermal energy supply and demand in time and space, which have received unprecedented attention in the past few years. To effectively address the undesirable inherent defects of pristine PCMs such as leakage, low thermal conductivity, supercooling, and corrosion, enormous efforts have been dedicated to developing various advanced microencapsulated PCMs (MEPCMs).

phase change microcapsules low-dimensional nanofillers thermal conductivity

1. Core Materials of MEPCMs

Phase change materials (PCMs) is a kind of typical latent heat thermal energy storage (TES) material with the advantages of large heat storage density, operational simplicity, and high safety, which can control the temperature by reversibly absorbing or releasing thermal energy to the environment [1]. Thus, PCMs, as an excellent storage media, have a wide range of applications, including solar energy systems, buildings, and thermal management of vehicles battery and electronic devices [2][3][4][5][6][7][8]. Generally, PCMs can be divided into solid-solid, solid-liquid, solid-gas, and liquid-gas PCMs according to their phase transition states [9]. Although the latent heat of solid-gas and liquid-gas PCMs is larger than that of solid-liquid and solid-solid PCMs, it is difficult to be applied in engineering fields due to their great volume change in phase transition process. Besides, the solid-solid PCMs which stored heat by changing crystal form, have a relatively smaller phase transition enthalpy than solid-liquid PCMs [10]. Correspondingly, the solid-liquid PCMs have unique advantages in phase equilibrium, volume change and vapor pressure. Thus, the solid-liquid PCMs become the most significant core materials employing in phase change microcapsules [11]. When the external temperature is higher than the solid-liquid PCMs, the solid-liquid PCMs will absorb heat in the form of sensible heat, and the temperature begins to rise. When the phase transformation temperature is reached, the temperature will remain constant. This heating process can effectively store energy in the form of latent heat until it is completely converted into liquid and then raising the temperature in the form of sensible heat (and vice versa). The cooling process also has a period of releasing energy in the form of latent heat.

Solid-liquid PCMs can be further divided into organic, inorganic, and eutectic PCMs according to their composition [12]. Among them, organic PCMs have the characteristics of wide applicable temperature range and stable chemical properties, which mainly include paraffins, fatty acids, esters, and alcohols [13]. Typically, paraffins, as the most common commercial organic PCMs, contain a variety of high-purity straight chain alkanes, and their melting temperature and melting enthalpy can increase with the number of carbon atoms increasing. Compared with high-purity straight chain alkanes, paraffins are usually produced as the by-products in crude oil refining. Thus, they are widely available and inexpensive, and have higher practical application value [14][15]. Fatty acid is another organic PCMs produced from common vegetable oils and animal oils, whose cost is higher than that of paraffin. The commonly used fatty acids include stearic acid (SA) (melting point 69 °C), palmitic acid (melting point 56 °C), myristic acid (melting point 58 °C) and lauric acid (melting point 49 °C), etc. Due to their suitable phase transition temperature and high phase transition enthalpy, fatty acids are the most promising non-paraffin PCMs. Besides, sugar alcohol can also be used as PCMs, whose melting temperature and melting enthalpy are the highest among organic PCMs, so it has good application prospects in solar heating and industrial waste heat recovery. Notably, though organic PCMs have various advantages, almost all of them belong to low-temperature PCMs with a melting point lower than 220 °C [16]. Moreover, their thermal conductivity is relatively low, and usually suffer from high flammability and low thermal stability. Different from organic PCMs, the inorganic PCMs exhibit a relatively higher thermal conductivity and greater phase change enthalpy. They also have a clear melting point, excellent flame resistance, and good recyclability. Inorganic PCMs can be roughly divided into hydrated salts and molten salts. When the PCMs are hydrated salts, the solid-liquid phase change process is actually the dehydration and hydration process of hydrated salts, such as $MgSO_4 \cdot 7H_2O$ (melting point 49 °C), $CaCl_2 \cdot 6H_2O$ (melting point 33 °C), $Ba(OH)_2 \cdot 8H_2O$ (melting point 78 °C), $Al(OH_3)_2 \cdot 7H_2O$ (melting point 75 °C). Usually, two or more hydrated salts are mixed to adjust the phase transition temperature to meet the requirements of practical applications. It is mainly suitable for medium and low temperature TES since the melting point of most hydrated salts is below 220 °C, while molten salts such as sulfate and nitrate can run at a temperature of more than 420 °C and can be used in high temperature TES applications [17][18]. Eutectic PCMs can be prepared by inorganic-inorganic, organic-organic or two kinds of mixed PCMs, which can achieve high thermal conductivity, small supercooling, and a relatively low cost through adjusting their specific proportion. Eutectic PCMs have a wide range of melting points and can be used in low, medium, and high temperature applications. Most organic-inorganic eutectic PCMs belong to low temperature PCMs, such as $CO(NH_2)_2$-NH_4Br (melting point 80 °C), while most inorganic-inorganic eutectic PCMs belong to medium temperature PCMs with melting point between 220 °C and 420 °C [19][20]. Their advantages, disadvantages, and the melting points of typical organic, inorganic, and eutectic PCMs are summarized in **Table 1**.

Table 1. Advantages, disadvantages and melting points of typical PCMs.

Type	PCMs	Advantages	Disadvantages	Melting Point (°C)
Organic	Paraffin C16-C18	Wide applicable temperature range Less supercooling phenomenon High crystallization rate High chemical stability Strong recycling performance Safety and non-toxic	Low thermal conductivity Poor thermal storage capacity per unit volume Volatilize easily Flammability Expensive	22
	Paraffin C20-C33			50
	Stearic acid			69
	Palmitic acid			56
	Myristic acid			58

Type	PCMs	Advantages	Disadvantages	Melting Point (°C)
	Lauric acid	Little corrosion performance		49
Inorganic	$MgSO_4 \cdot 7H_2O$	Lower volumetric expansion Large heat storage capacity Low cost High thermal conductivity Greater phase change enthalpy Non-flammable With a clear melting point Recyclable	High supercooling Prone to precipitation Poor dimensional stability Low thermal stability Corrosive Phase segregation Poor compatibility with some building materials	49
	$CaCl_2 \cdot 6H_2O$			33
	$Ba(OH)_2 \cdot 8H_2O$			78
	$Al(OH_3)_2 \cdot 7H_2O$			75
	KNO_3			340
	Na_2CO_3			850
Eutectic	$CO(NH_2)_2$-NH_4Br	High thermal conductivity Hardly segregation Hardly supercooling	Expensive Strong odor	80
	$Na(CH_3COO) \cdot 3H_2O$-$CO(NH_2)_2$			30
	$NaCO_3$-$LiCO_3$			490
	NaF-MgF_2			640

The shell of phase change microcapsules can effectively prevent the leakage of PCMs during the phase change process. Obviously, to maintain the structural stability of microcapsules, the shell materials need to have high encapsulation efficiency, excellent thermal and chemical stability, and superior compatibility. In the past few decades, extensive efforts have been dedicated to enhance the comprehensive properties of microencapsulated PCMs (MEPCMs) by the reasonable design of shell materials [21]. Usually, these shell materials can be divided into organic, inorganic, and organic-inorganic hybrid materials according to their chemical properties.

2.1. Organic Shells

Organic shell materials are generally composed of natural and synthetic polymer materials which have good elasticity, toughness, excellent compactness, and stable chemical properties. Typically, melamine-formaldehyde (MF) resin and urea-formaldehyde (UF) resin are the most reported and widely used organic shell materials of microcapsules [22][23][24][25][26][27][28][29]. For example, Yu et al. designed a type of MEPCMs by using MF as the shell material and dodecanol as the core material via in-situ polymerization, achieving the highest phase transition enthalpy of 187.5 J/g and a high encapsulation efficiency of 93.1% [28]. Compared with MF, UF has higher phase transition latent heat and relatively poor thermal resistance [30][31][32][33][34][35][36]. Thus, Tohmura et al. developed melamine-urea-formaldehyde resin (MUF) by the simultaneous reaction of cyanuric acid and urea with formaldehyde to effectively improve the intermolecular force, thereby improving its thermal stability [36]. Besides, polymethylmethacrylate (PMMA) is also a common shell material for MEPCMs in recent years due to its environmental stability, and low easy processing cost [37][38][39][40][41]. However, the residual small molecules (such as formaldehyde, acrylate, etc.) of the above resins pose a threat to the environment and human health. Therefore, more and more studies have been devoted to the development of polyurea or polyurethane (PU) encapsulated MEPCMs [42][43][44]. In particular, the initial reaction rate between diisocyanate and polyol is relatively lower, and the elasticity of MEPCMs shell can be improved by adjusting the soft segment structure of PU, which is very beneficial to the mechanical properties and compactness of MEPCMs [45]. Therefore, in the previous study, one team has realized the microencapsulation of methyl laurate with PU shell through interfacial polymerization [46][47][48]. In addition, some polymers including polystyrene (PS) [49][50][51][52][53] and starch [54], etc are also used as organic shell materials to prepare MEPCMs. However, it is noting that most organic materials face the challenges of high flammability and low thermal conductivity.

2.2. Inorganic Shells

Compared with the organic polymers, the inorganic shell materials have higher thermal conductivity and superior mechanical strength, which can not only improve the durability and reliability of the microcapsules, but also improve the heat transfer performance of MEPCMs. Therefore, the use of inorganic materials as microcapsules shells has become another significant trend [21]. Common inorganic shell materials include silica oxide (SiO_2) [55][56][57][58][59][60][61][62][63][64][65][66][67][68][69][70][71], calcium carbonate ($CaCO_3$) [72][73][74][75][76], graphene [77][78][79], titanium dioxide (TiO_2) [80][81][82][83], etc. Among them, SiO_2 is a commonly used shell due to its wide source, low cost, and mature preparation method. For example, Liang et al. developed n-octadecane-based MEPCMs with silica shell by interfacial polymerization, and the phase change enthalpy and encapsulation rate of the microcapsules were as high as 109.5 J/g and 51.5%, respectively [56]. Besides, the graphene with superb intrinsic thermal conductivity can significantly improve the thermal conductivity of MEPCMs and provide a new material for thermal energy management [79]. In addition, metal oxides have also been widely used as an inorganic shell, including TiO_2, Al_2O_3, Fe_3O_4, and ZnO, etc [84]. For example, Liu et al. synthesized n-eicosane MEPCMs with TiO_2 as shell by interfacial polymerization, and found that the obtained microcapsules not only had durability and highly stable shape, but also exhibited superior antibacterial function, showing great potential in medical applications [82]. However, it is undeniable that the inorganic shell materials inevitably suffer from the disadvantage of poor stability and brittleness.

2.3. Organic-Inorganic Hybrid Shells

Organic-inorganic hybrid shells can combine the high inherent thermal conductivity of inorganic materials and the toughness of polymer shells, and achieve a high thermal conductivity, permeability resistance and thermal stability of MEPCMs. Therefore, the organic-inorganic hybrid shell exhibit promising potential in next-generation shell materials of MEPCMs [21][85]. It can be expected that the incorporation of inorganic nanofillers in organic shells can endow MEPCMs with high thermal conductivity and some unique properties [86][87][88][89][90][91]. Typically, Pickering emulsion polymerization is a classical method to obtained the polymer/inorganic hybrid shells. For example, Yin et al. prepared n-hexadecanol MEPCMs using MF-SiO_2 as the hybrid shell by Pickering emulsion polymerization [86]. The SiO_2 particles in the shell significantly improved the mechanical strength and thermal reliability of the MEPCMs, and received a high phase change enthalpy of 163.76 J/g. Furthermore, Zhang et al. synthesized a unique graphene oxide (GO)/polyaniline (PANI) hybrid shell through emulsion polymerization, which combined the excellent barrier properties of GO with the anti-corrosion function of PANI. The results show that the MEPCMs have strong solvent resistance to organic solvents, and the MEPCMs are dispersed in waterborne epoxy resin, which can prepare intelligent coatings with dual functions of anti-corrosion and self-healing [92]. In short, the MEPCMs with polymer-inorganic hybrid shells have become the current research focus of phase change microcapsules, which hold great potential in thermal management applications. **Table 2** summarizes the phase change enthalpy and encapsulation rate of MEPCMs encapsulated by some representative shell materials.

Table 2. Typical shell materials in the preparation of MEPCMs.

Shell Type	Shell Materials	Core Materials	Encapsulation Methods	Phase Change Enthalpy (J/g)	Encapsulation Rate (%)	Refs.
Organic shells	MF	Lauryl alcohol	In-situ polymerization	187.5	93.1	[28]
	UF	Paraffins	In-situ polymerization	72.4	52.8	[32]
	PU	Methyl laurate	Interfacial polymerization	136.2	-	[46]
	PMMA	$Na_2HPO_4 \cdot 12H_2O$	Solvent evaporation	142.9	80.4	[41]
	Polyurea	$CaCl_2 \cdot 6H_2O$	Interfacial polymerization	118.3	71	[43]
	PS	N-tetradecane	In-situ polymerization	98.71	44.7	[53]
	Starch	N-heptadecane	Interfacial polymerization	187.27	78.27	[54]
Inorganic shells	SiO_2	N-octadecane	Interfacial polymerization	109.5	51.5	[56]
		Mannitol	Sol-gel	252.66	89.6	[69]
	$CaCO_3$	N-tetradecane	Self-assembly	58.54	25.86	[74]
	rGO	SA	Pickering emulsion polymerization	159	74.3	[79]
	TiO_2	N-eicosane	Interfacial polymerization	188.27	-	[82]

Shell Type	Shell Materials	Core Materials	Encapsulation Methods	Phase Change Enthalpy (J/g)	Encapsulation Rate (%)	Refs.
Organic-inorganic hybrid shells	MF-SiO_2	N-hexadecanol	Pickering emulsion polymerization	163.76	74.6	[86]
	PMMA-BN/TiO_2	Paraffin	Pickering emulsion polymerization	124.4	72.1	[87]
	P(MMA-co-BA)-TiO_2	Paraffin	Pickering emulsion polymerization	90.12	36.09	[88]
	PUA-TiO_2	N-octadecane	Interfacial polymerization	181.1	77.3	[89]
	PS-GO	N-hexadecane	Pickering emulsion polymerization	186.8	78.5	[91]

e problem during the phase change process, but also improve the thermal conductivity and other properties of PCMs, which shows a good development prospect in many applications. Most of MEPCMs are spherical with a particle size from 1 to 1000 μm, and their mass of PCMs cores vary from 20% to 95% in the total mass of MEPCMs [93]. Typical preparation strategies of the MEPCMs were described as follows. It can be mainly divided into physical method, chemical method, and physical-chemical method according to the synthesis mechanism.

3.1. Physical Method

In general, the physical methods for preparing MEPCMs mainly include spray drying and solvent evaporation [94][95]. The spray drying method is that the core material and the shell material are co-dissolved in a solvent, and the mixture is sent into a heating chamber in the form of small droplets for heating. During the heating process, the solvent will evaporate, and finally the microcapsules are separated. For example, Borregro et al. microencapsulated the paraffin by spray drying, and the encapsulation efficiency reached 63%. Besides, they found that the properties of these MEPCMs were closely related to where they were collected in the spray dryer, and the MEPCMs was highly stable and reversible even after 3000 cycles [95]. Overall, the spray drying method is relatively simple to operate and has a high production efficiency, but it is not suitable for inorganic PCMs [96]. Another physical synthesis method is the solvent evaporation method [97]. Specifically, the polymer shell materials were dissolved in volatile solvent, and then adding PCMs into above solution to form emulsion, finally the shell is formed on the droplet surface by evaporating the solvent.

3.2. Chemical Method

Chemical method is the most widely used method to prepare MEPCMs. It usually uses free radical polymerization to form oil-in-water or water-in-oil emulsions, and reacts on the oil/water (O/W) interface to form shell materials. The representative and commonly used chemical methods include in-situ polymerization, interfacial polymerization, suspension polymerization and emulsion polymerization. Typically, the in-situ polymerization method is to emulsify the core material to form oil-in-water emulsion droplets under the action of emulsifier, and then the obtained O/W emulsion droplets were catalytically polymerized with monomers or prepolymers under certain conditions to form a polymer shell, and finally cured to form MEPCMs. Generally, most of MEPCMs with MF and UF shells are synthesized by in-situ polymerization. Notably, this synthesis method can achieve the synergetic integration of high encapsulation efficiency and uniform coating, but the operation process is relatively complex and may cause some environmental pollution [98]. Another chemical method for preparing MEPCMs is interfacial polymerization. In the interfacial polymerization, the core material and hydrophobic monomer are used as the oil phase, while the emulsifier and deionized water serve as the water phase, which are emulsified to form an oil-in-water emulsion, and finally polymerize at the O/W interface to form a shell under appropriate conditions. Zhang et al. synthesized a n-octadecane-based MEPCMs with a polyurea shell by interfacial polymerization. It was found that when the core-shell ratio was 7/3, the MEPCMs exhibited better phase transition performance, better permeation resistance and higher encapsulation efficiency [99]. In general, interfacial polymerization is easy to operate and low cost, but the reaction speed is often too fast, resulting in the properties of final product is more difficult to control [100]. Besides, suspension polymerization is to mix the PCMs, reaction monomers and initiator as the oil phase, which is then suspended in the water phase to form emulsion droplets under the action of surfactants, finally polymerize to form MEPCMs [101]. Typical MEPCMs with organic shell materials such as PMMA and PS are usually prepared by suspension polymerization. Compared with above polymerization methods, the suspension polymerization usually has a higher encapsulation rate and strong thermal regulation ability, but the production cost is also relatively higher [102]. In addition, the emulsion polymerization method is to mix the PCMs with reaction monomers, and add emulsifiers to form the O/W emulsions, finally initiate polymerization to form microcapsules under the action of initiator [103][104]. Particularly, Pickering emulsion polymerization has been widely applied to prepare MEPCMs in recent years due to the superior mechanical properties and thermal stability of as-obtained MEPCMs. Moreover, Pickering emulsion polymerization is more environmentally friendly and easy to obtain polymer/inorganic hybrid shells [102][105]. Wang et al. prepared a MEPCMs with Pickering emulsion polymerization by introducing modified SiO_2 and TiC nanoparticles as emulsifiers [106]. The obtained MEPCMs exhibited well-defined core-shell structure, and have good heat storage and release properties, superior thermal stability, and enhanced thermal conductivity. So far, Pickering emulsion polymerization has become an important part of the methods for preparing of advanced MEPCMs due to its simplicity and economy.

3.3. Physical-Chemical Method

Physical-chemical methods are usually used to prepare microcapsules under external force and chemical reaction by combining physical processes such as heating and cooling with chemical processes such as crosslinking and condensation. Coacervation is a typical and commonly used physical-chemical method, which can be further divided into single coacervation and complex coacervation method [107][108]. For example, complex coacervation method is to form emulsion by mixing PCMs with a polymer, then emulsifying with another polymer to form stable emulsion, and finally condensing to form MEPCMs. Sol-gel method is to hydrolyze the reaction monomer to form sol solution, and then mix it with the PCMs to form emulsion [109][110]. Under certain reaction conditions, the gel shell is generated around the PCMs droplet through polycondensation reaction [111].

In summary, there are many encapsulation methods that can be used to prepare MEPCMs. However, as shown in **Table 3**, different preparation methods all have some advantages and disadvantages. For example, although physical methods are simpler and more economical, the prepared MEPCMs are usually agglomerated together. Moreover, Pickering emulsion polymerization is particularly suitable for the preparation of MEPCMs with organic-inorganic hybrid shells, which is of great significance for achieving the enhanced thermal conductivity and functionalization of MEPCMs, but still faces some challenges for large-scale implementation. Therefore, in practical applications, the selection of an encapsulation method not only depends on the performance and parameters of MEPCMs (e.g., size, size distribution, chemical stability, and thermal stability), but also is inextricably linked to the polymerization operability, cost, etc.

Table 3. Comparison and summary of the different preparation methods of MEPCMs.

Types	Methods	Advantages	Disadvantages	Scope of Applications
Physical method	Spray drying	Simple operation High production efficiency Wide range of sizes	Low packaging rate Not used for inorganic PCMs	Organic PCMs Heat sensitive material
	Solvent evaporation	Low cost	Low encapsulation efficiency	Inorganic PCMs
Chemical method	In-situ polymerization	High encapsulation efficiencyStable shape Uniform coating	Complex operation Harmful for the environment	Organic PCMs Inorganic PCMs Organic shell material such as MF and UF
	Interfacial polymerization	High reaction speed Simple operation Low permeability	The monomer is required to have a high reactivity Harmful for the environment	Organic PCMs Inorganic PCMs Organic shell material such as UF
	Suspension polymerization	Environmentally friendly Facile reaction condition High packaging rate	High energy consumption Expensive Not used for inorganic PCMs	Organic PCMs Large grained Organic shell material such as PMMA
	Emulsion polymerization	Stable High packaging rate High preparation efficiency Environmentally friendly	Complicated Expensive	Organic PCMs Inorganic PCMs Organic shell material such as PMMA Polymer/inorganic hybrid shell
Physical-chemical	Coacervation	Simple equipment Good control of the	Difficult to scale-up Not used for inorganic	Organic PCMs Organic shell material

Types	Methods	Advantages	Disadvantages	Scope of Applications
method		particle size and thickness	PCMs Agglomeration	
	Sol-gel method	High thermal stability Strong controllability	Non-insulated and limited in building applications	Organic PCMs Inorganic PCMs Inorganic shell material

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