

Preparation and Property Analysis of Phase Change Concrete PEG/SiO₂-CPCM

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ABSTRACT

With polyethylene glycol (PEG) as the phase change material (PCM), and silica sol as inorganic porous carrier, this paper prepares the polyethylene glycol/silica sol composite phase change material (PEG/SiO₂-CPCM) using sol-gel method, and adds PEG/SiO₂-CPCM into concrete to prepare phase change concrete according to the absolute volume method. Next, this paper uses scanning electron microscopy (SEM) and differential scanning calorimetry (DSC) to characterize the microstructure, compatibility, stability and energy storage of PEG/SiO₂-CPCM, and tests the compressive strength and thermal conductivity of the phase-change concrete. The results show that PEG-2000 had a good compatibility with SiO₂; the mass fraction of PEG in the CPCM should be controlled within 90 %; after 200 thermal cycles, the mass loss is less than 1 %. The PEG/SiO₂-CPCM starts to undergo phase change at the temperature of 42.4 °C; the strength of the phase-change concrete with a 5 % PEG content dropped by about 4.96 %; under the premise of ensuring good working performance, the addition of the PEG/SiO₂-CPCM can greatly enhance the thermal conductivity of the concrete.

1. INTRODUCTION

Concrete is a kind of material with poor thermal conductivity. Under the influence of sunlight, there is often a large temperature difference between the inside and the outside of the box section, which produces significant temperature difference stress and results in a large temperature difference tensile stress. As the tensile strength of the concrete structure is only 2-3 Mpa, the concrete box structure often has surface cracks due to the large temperature difference between the inside and outside of the section. The existing research results show that when the temperature difference between the inner and outer surfaces of the concrete box is greater than 10 °C, the temperature stress may be greater than 2.5 MPa. As long as measures are taken to reduce the temperature difference between the inner and outer surfaces to a certain extent, the occurrence of concrete cracks could be avoided [1, 2].

Phase Change Materials (PCMs), also known as latent thermal energy storage materials (LTES), utilize the heat absorbed or released during the phase change process to keep the temperature of the material itself relatively stable, and it has the functions of heat storage and temperature regulation [3]. Therefore, in the fields of building energy conservation, engineering structure cooling, road maintenance, *etc.*, people add PCMs to concrete by direct mixing or encapsulating in the hopes of obtaining phase-change concrete with “self-temperature regulation effect” [4-7]. There are various PCMs, but those suitable for construction engineering are often solid-liquid PCMs such as paraffin and aliphatic acids. Such PCMs generally have the advantages of suitable phase change temperature, high phase change enthalpy, low price and no supercooling [8-10].

PEG has received wide attention due to its advantages of wide phase change temperature range, high latent heat of phase change, good thermal stability, no phase separation, low degree of supercooling, safe and non-toxic, no volatility, low corrosivity, and stable chemical properties, etc. However, in terms of phase-change patterns, PEG is a kind of organic solid-liquid phase change heat storage material, that is, during the phase-change process, flowing liquid will be generated and it's easy to leak when in use, which has greatly limited its practical applications [11-13]. Silica sol is an ideal inorganic porous carrier. It has a rich three-dimensional network porous structure after gelation, and large pore volume and specific surface area. It can firmly adsorb the phase change heat storage materials into its microporous structure, and its action with the phase change heat storage material is only a physical interaction, which does not damage the heat storage performance of the phase change heat storage material; moreover, it has good shaping effect, wide sources, low price, safety and non-toxicity. Therefore, this study adopts silica sol to mix with PEG-2000 to prepare the PEG/SiO₂ CPCM.

This paper prepares PEG/SiO₂-CPCM using sol-gel method, and tests its microstructure, thermal properties, and cyclic phase change stability, at last, it adds PEG of different proportions to the CPCM to study its influence on the strength and thermal conductivity of the concrete.

2. EXPERIMENT

2.1 Raw materials

PEG is a commonly used polyether phase change heat storage material, its molecular structure is consisted of long-

chain polymers with hydroxyl groups at both ends, and it has good hydrophilicity. According to different polymerization degrees, a series of polymers with average molecular weights ranging from 200 to 20,000 can be formed. Through experiments it's found that PEG with a molecular weight of 2000 is most suitable for engineering structure related applications [16]. PEG used in this experiment is produced by Tianjin Guangfu Fine Chemical Research Institute and is provided by Shanghai Macklin Biochemical Co., Ltd, analytically pure, its phase change start temperature is about 38.2 °C, peak temperature is 52.2 °C, end temperature is 72.2 °C, and the phase change enthalpy reaches 185.8 J/g.

Silica sol: industrial grade, produced by Zhejiang Yuda Chemical Co., Ltd., mass content 30 %, particle size 11.7 nm, pH value 9.9, density 1.198 g/cm³ at 20 °C.

2.2 Preparation of PEG/SiO₂ composite shape-stabilized phase change material

This paper adopted the sol-gel method. At room temperature, PEG of a certain mass ratio was added into the silica sol solution, the mass ratio of the PEG and the silica sol is shown as Table 1.

Table 1. Mass ratio of PEG and silica sol

Composite shape-stabilized phase change material	1#	2#	3#	4#	5#	6#	7#	8#
Percentage (%)	0	30	40	50	60	70	80	90
PEG Mass (g)	0	12.9	20	30	45	70	120	270
Silica Sol Mass (g)	100	100	100	100	100	100	100	100

The mixture of PEG and silica sol was thoroughly mixed with a glass rod or electric stirring paddle until the PEG flakes or powders completely disappeared, so that the PEG was uniformly dispersed in the silica sol; then calcium chloride solution of a certain concentration was added into the mixture and stirred constantly until the mixture was completely gelled. The preparation process is shown in Figure 1.

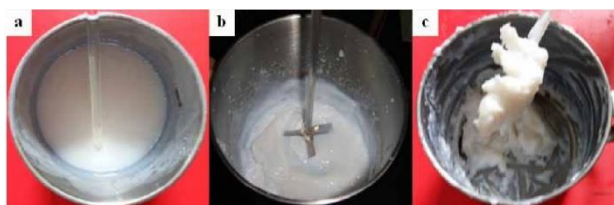


Figure 1. Preparation flow of PEG/SiO₂ composite shape-stabilized phase change material

In the preparation process of PEG/SiO₂ composite shape-stabilized phase change material, when the mass fraction of PEG reached 90 % (8# mass ratio), the PEG/SiO₂ composite shape-stabilized phase change material was dried at room temperature and then formed pie-shaped lumps and the surface was sticky; When heated, the material presented a semi-flow state, and there were some liquid PEG exuded from edges and corners, indicating that the mass fraction of PEG was too high and the microporous capacity of the SiO₂ gel had reached saturation, the excess PEG molecules formed a coating on the surface of the SiO₂ gel, since there's no binding force, the PEG molecules were heated and underwent solid-liquid phase change, producing flowing liquid phase, which affected the shaping effect. In the two coagulation methods, the phenomenon occurred in the 8# mass ratio, while for 1#~7# mass ratios, PEG liquid exudation did not occur. Therefore, it can be preliminarily judged that the mass fraction of PEG in PEG/SiO₂ composite shape-stabilized phase change material should be lower than 90 %.

2.3 Test and characterization methods

By comparing and observing the appearance of PEG/SiO₂-CPCM of different ratios and judging whether there're lumps on the surface, the saturated adsorption rate could be determined, and then SEM (model: KYKY-2800B, produced

by Gold APP Instrument Corporation China) was used to analyze its surface morphology and microstructure and study the adsorption of PEG-2000 by SiO₂.

The structural characteristics of PEG/SiO₂-CPCM were characterized by an infrared spectrometer (model: Spectrum 100, produced by PerkinElmer U.S.), the compatibility of PEG-2000 and SiO₂ was analyzed. The sample preparation method was KBr pellets method, and the scanning frequency was 3 times.

The phase change temperature and latent heat of phase change of PEG-2000 and PEG/SiO₂-CPCM were tested by a thermal gravimetric analyzer (model: STA449C, produced by NETZSCH, Germany), the scanning temperature of the instrument was 20 °C~130 °C, and the temperature heating/cooling rate was 2 °C/min. Both the sweep gas and protective gas were high purity nitrogen with a gas flow rate of 20 mL/min. First, temperature rose from 20 °C to 130 °C, the water was evaporated and the influence of historical temperature was eliminated, then the temperature dropped from 130 °C to 20 °C, the temperature was kept for 2 min, and then the temperature rose from 20 °C to 130 °C again.

A certain amount of PEG/SiO₂-CPCM particles were naturally deposited on the filter paper, then placed in a vacuum drying oven to undergo 20 °C-80 °C phase change thermal cycle for 10, 20, 50, 100, and 200 times, and the mass loss situation was recorded.

For the compressive strength test, the test pieces were cubic blocks with a size of 100×100×100 mm, 6 blocks in each group, cured for 3d, 7d and 28d respectively, and then subject to the test using WE-1000 hydraulic universal testing machine according to the requirements of GB/T 50081—2002 *Standard for Test Method of Mechanical Properties on Ordinary Concrete*, the compressive strength test loading rate was 5 kN/s, the indoor temperature was 25 °C, and the loading rate was uniform and continuous.

The thermal conductivity of concrete with different mixing ratios was tested by a smart plate thermal conductivity tester (model: IMDRY600-II). The size of the test pieces was 600×600×50 mm, and the number of the test pieces was the same as the ratio of the cubic blocks. Before the test, the test pieces were put in the oven for 48 h, the test time was 6 h, and the sampling interval was 10 min. The two states of normal temperature and high temperature phase change were tested respectively. Under the normal temperature state, the cold

plate temperature was 15 °C, the hot plate temperature was 35 °C, under the high temperature phase change state, the cold plate temperature was 40 °C, and the hot plate temperature was 60 °C.

2.4 Design of phase change concrete mixing ratios

The test pieces were cubic blocks with a size of 100×100×100 mm, the concrete volume for the on-site material fabrication was 0.01 m³. The particle size of larger pebbles was 10~20 mm, and the particle size of smaller pebbles was 5~10 mm, the mixing ratio was 6:4. All pebbles were sieved to reduce the content of mineral powder, and the

apparent density of the pebbles was 2734.1 kg/m³; the sand was medium sand with an apparent density of 2635 kg/m³ and a modulus coefficient of 2.83, the water content is negligible; For cement, the ordinary Portland cement 42.5 R (brand: Xiaonanhai) was adopted; the water reducing agent was high-efficient polycarboxylate water reducer with a water reduction rate of 20 %-25 %, the recommended content was 0.8 %~1.2 %; after the dried gel was ground and sieved, powders of the composite shape-stabilized phase change material with a particle size less than 0.6 mm can be obtained, namely the PEG/SiO₂-CPCM, and its apparent density could reach 1.39 g/cm³.

Table 2. Mixing ratio of standard concrete

Water cement ratio	Water /kg	Cement /kg	Medium sand /kg	Pebbles /kg	Fly ash /kg	Water reducing agent /kg
0.35	148	420	720	1080	100	6.24

Table 3. Mixing ratio of phase change concrete

No	Water	Cement	Medium sand	Pebbles	Fly ash	Water reducing agent	PCM volume ratio
P-0	148	420	720	1080	100	6.24	0%
PEG-V5	148	420	720	1080	100	6.24	5%
PEG-V10	148	420	720	1080	100	6.24	10%
PEG-V10	148	420	720	1080	100	6.24	15%

The mixing ratio of phase change concrete adopted the absolute volume method, and the phase change aggregates were directly added to the mixture according to the volume fraction ratio. A total of 4 groups of comparative experiment were designed for common concrete and PEG/SiO₂-CPCM phase change concrete with a volume fraction of 5 %~15 %, the mixing ratio of the phase-change concrete is shown in Table 3, the preparation process of the concrete is shown in Figure 2.



Figure 2. Preparation of concrete test pieces

3. RESULTS AND DISCUSSION

3.1 PEG/SiO₂ adsorption rate and SEM analysis

Figure 2 shows SEM graphs of PEG-2000 and PEG/SiO₂-CPCM. It can be clearly observed that the SiO₂ gel has a rich three-dimensional network porous structure, with large specific surface area and strong adsorption capacity; the PEG molecules present continuous and uniform sheet-shaped structures and can be embedded into the three-dimensional network structure by sol-gel interaction. From the right graph we can see that, the PEG/SiO₂ still maintains the three-dimensional network structure, a large number of voids have been filled by PEG-2000 molecules, and the surface gradually becomes smooth, but no lumps or agglomerated PEG-2000 molecules were exposed or existed. Therefore, the PEG/SiO₂-CPCM prepared by the sol-gel method can ensure a high

adsorption rate and good encapsulation effect.

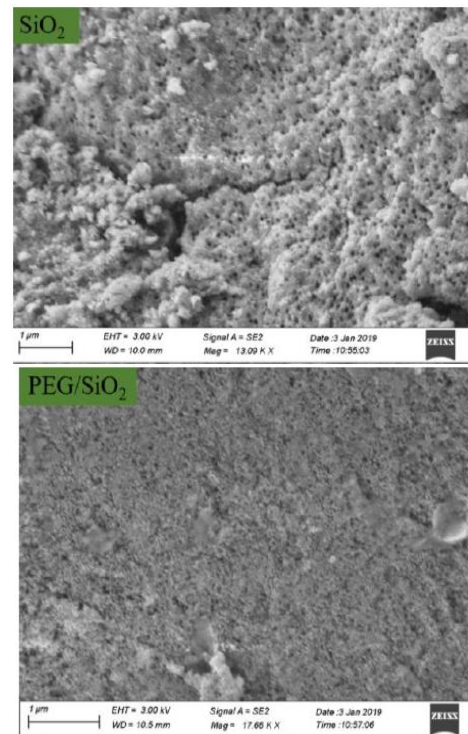


Figure 3. SEM graphs of SiO₂ and PEG/SiO₂

3.2 Compatibility analysis

Figure 4 shows infrared spectrum graphs of PEG-2000, SiO₂ and PEG/SiO₂-CPCM. It can be seen from Figure 4 that 3343.05 cm⁻¹ is the -OH antisymmetric stretching vibration peak, which is slightly red shifted compared with pure PEG-2000, this is due to the formation of hydrogen bonds between the C-OH of PEG and the Si-OH of the SiO₂ gel; The peaks at

2888 cm^{-1} and 1468 cm^{-1} were C-H stretching vibration absorption peak and C-H bending vibration absorption peak, respectively; the peak at 1344 cm^{-1} was -OH in-plane bending vibration absorption peak; and the absorption peaks at 1114 cm^{-1} and 962 cm^{-1} were C-O antisymmetric stretching vibration peak and C-O symmetric stretching vibration peak, respectively; the absorption peak at 842 cm^{-1} is the -CH₂ bending vibration peak, all above mentioned peaks are characteristic absorption peaks of PEG molecules. The crab foot-shaped absorption peak at 800~1500 cm^{-1} is the C-O-C bending vibration peak, because the O in the C-O-C forms a hydrogen bond with the -OH on the inner surface of the SiO₂ gel, so compared with the corresponding peak positions of PEG, a red shift has occurred and the peak intensity is also slightly weakened. Comparing the infrared spectrum graphs of PEG-2000, SiO₂ and PEG/SiO₂-CPCM, we can see that for the three materials, no obvious new characteristic peak has appeared, and no obvious characteristic peak has disappeared, therefore, the interaction between PEG-2000 and SiO₂ is pure physical adsorption, no chemical reaction occurred during the contact of the two, and the compatibility was good.

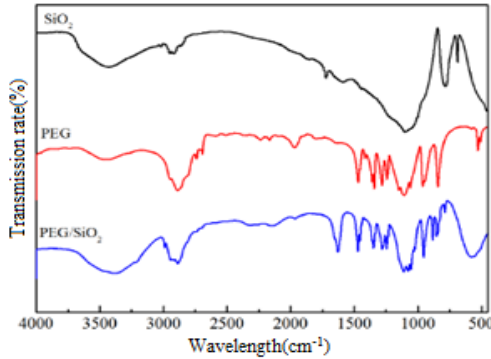


Figure 4. Infrared spectrum graphs of PEG, SiO₂ and PEG/SiO₂-CPCM

3.3 DSC analysis

Figure 5 shows the DSC curves of PEG-2000 and PEG/SiO₂-CPCM. The results show that, in the heating process, the phase change start temperature and peak temperature of PEG/SiO₂-CPCM are about 2 °C higher than

PEG-2000, their melting intervals are basically the same; the latent heat of phase change of PEG/SiO₂-CPCM is significantly lower than that of pure PEG-2000; in the cooling process, the phase change characteristics of PEG-2000 and PEG/SiO₂-CPCM are basically the same.

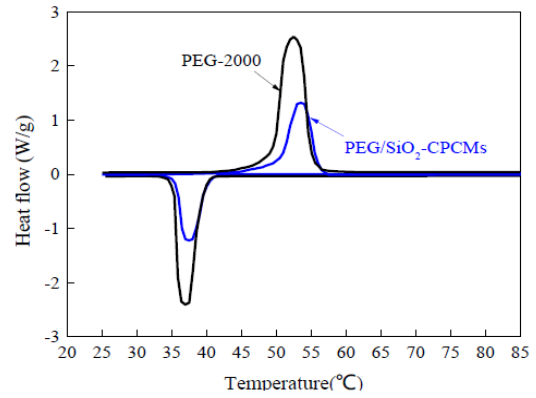


Figure 5. DSC curves of PEG and PEG/SiO₂-CPCM

Since the combination of PEG-2000 and SiO₂ is purely physical, the phase change enthalpy of PEG/SiO₂-CPCM is theoretically linear with the mass fraction of PEG-2000, their relationship can be expressed by Formula (1):

$$\Delta H_t = \omega_{PEG} \cdot \Delta H_{PEG} \quad (1)$$

where ΔH_t is the theoretical enthalpy, ω_{PEG} is the mass fraction of PEG, ΔH_{PEG} is the measured enthalpy.

Table 4 lists the thermal parameters of PEG-2000 and PEG/SiO₂-CPCM. Using Formula (1), it can be calculated that, in the heating process, the theoretical enthalpy of PEG/SiO₂-CPCM is about 121.03 J/g, the measured enthalpy is 82.1 J/g, and the phase change enthalpy is reduced by 38.93 J/g. Through analysis, it is believed that the coexistence of SiO₂ gel and Na⁺ as an impurity with PEG-2000 will hinder the crystallization performance of PEG-2000. In the gel structure, the ends of PEG-2000 molecules were confined, which has further limited the free condensation and crystallization of PEG-2000, thereby greatly reduces its phase change heat storage capacity [17].

Table 4. Thermal parameters of PEG and PEG/SiO₂-CPCM

No.	PEG content	Heating process				Cooling process			
		T_{on} (°C)	T_p (°C)	T_{end} (°C)	ΔH_m (J/g)	T_{on} (°C)	T_p (°C)	T_{end} (°C)	ΔH_c (J/g)
PEG-2000	100%	40.4	52.2	61.5	172.9	40.0	37.3	26.8	173.3
PEG/SiO ₂ -CPCMs	70%	42.4	54.6	65.2	82.1	39.4	34.2	26.3	75.9

Note: The symbol T_{on} in the table is the melting (crystallizing) start temperature, T_p is the melting (crystallizing) peak temperature, T_{end} is the melting (crystallizing) end temperature, ΔH_m is the melting enthalpy value, and ΔH_c is the crystallizing enthalpy value.

The PEG-2000 phase change start temperature is about 40.4 °C, the phase change end temperature is about 61.5 °C, and the peak value is about 52.5 °C; the PEG/SiO₂-CPCM phase change start temperature is about 42.4 °C, and the phase change end temperature is about 65.2 °C, the peak value is about 54.6 °C. In the fields of architecture, roads and structures, the structure surface temperature caused by atmospheric temperature field could reach as high as above 50 °C [18-20]. Therefore, when applying PEG/SiO₂-CPCM to concrete structures, it can fully undergo phase change under high

temperature conditions, thereby achieving the effects of energy storage and temperature regulation.

3.4 Phase change cycle stability

A certain amount of PEG/SiO₂ composite shape-stabilized phase change material particles were naturally deposited on a filter paper and placed in an oven at 200 °C for 2 h, and then subject to 20-80 °C thermal cycle for 10, 20, 50, 100, and 200 times. The phase change cycle stability of the PEG/SiO₂

composite shape-stabilized phase change material was investigated.

Table 5 shows the mass loss of PEG/SiO₂-CPCM after phase change treatment of different thermal cycles, the initial mass of the phase change material was 4 g. The experimental results show that the mass of PEG/SiO₂-CPCM decreased with the increase of the number of cycles, and the mass loss rate

tended to be stable. At the same time, after 200 times of thermal cycle treatment, the total mass loss rate of PEG/SiO₂-CPCM was less than 1 %. Therefore, the use of silica sol as a carrier can achieve a good encapsulation shaping effect, and can maintain a high retention rate after multiple thermal cycles, ensuring that the phase change enthalpy and heat storage capacity do not change significantly.

Table 5. Mass of PEG/SiO₂-CPCM after multiple phase change cycles

Cycle times	0	10	20	50	100	200
Mass (g)	4.0000	3.9834	3.9725	3.9734	3.9646	3.9631
Loss rate (%)	0.00	0.42	0.69	0.67	0.89	0.92

3.5 Mechanical properties of phase change concrete

Figure 6 shows the compressive strength values of phase-change concrete with different mixing ratios after cured for 3 d, 7 d and 28 d. The results show that the compressive strength of phase-change concrete decreased linearly with the increase of PEG/SiO₂-CPCM content, wherein the strength of PEG-V5 could reach the C50 grade requirement; while the strength of the 10 %~15 % content group was greatly reduced, but since the concrete in the PEG group showed good working performance, the concrete's strength increment mode was similar to that of the common concrete.

From Figure 7, it can be seen that with the increase of age, the strength of phase change concrete with different PEG/SiO₂-CPCM content decreased continuously, and the change of strength of phase change concrete of PEG-V5 group was the most obvious, the decline range gradually decreased from the 27.62 % of 3 d to only 4.96 % of 28 d; The strength of PEG-V10 group and PEG-V15 group phase change concrete decreased by more than 15 %, but for the phase change concrete with a 15% content, the compressive strength of 28 d still decreased by 57.07 %, reaching 22.4 MPa.

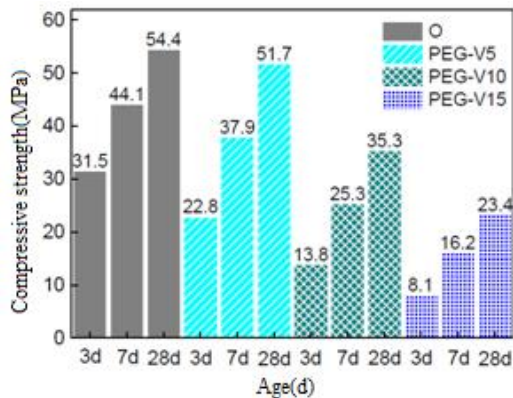


Figure 6. Phase change concrete compressive strength

Table 6. Thermal conductivity of phase change concrete (W/m·K)

No.	O	PEG-V5	PEG-V10	PEG-V15
Normal temperature test value	1.5316	1.5491	1.5270	1.6070
High temperature test value	1.3476	1.2417	1.1571	1.2439

4. CONCLUSION

(1) SiO₂ gel has a rich three-dimensional porous network structure and strong adsorption capacity, but the mass fraction

of PEG in the composite shape-stabilized phase change material should be less than 90%, and SEM and FT-RI test results indicate that the compatibility of PEG and SiO₂ is good, and no chemical reaction occurs. After 200 thermal cycles, the

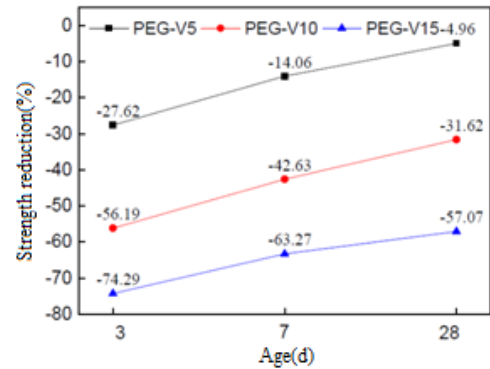


Figure 7. Strength change of phase change concrete

3.6 Thermal conductivity of phase change concrete

Table 6 lists the change law of thermal conductivity of common concrete and phase change concrete with a 5%~15% volume fraction content under the normal temperature and high temperature phase change conditions. The results show that at room temperature, the thermal conductivity of phase change concrete with 5% and 15% content is higher than that of common concrete, but the thermal conductivity of phase change concrete with 10% content is similar to that of common concrete. Through analysis, we believe that this is due to the decrease of the working performance of the concrete, the test pieces' pores and porosity increased, resulting in reduction in the thermal conductivity of the concrete plates; Under the high temperature conditions, the change law of the thermal conductivity is similar to that under the normal temperature conditions, but the thermal conductivity of the phase change concrete is reduced, through analysis, we believe that this is due to the PEG phase change in the concrete, a large amount of latent heat is absorbed, resulting in larger temperature differences between the cold plate and hot plate of the thermal conductivity instrument, so that the measured thermal conductivity becomes smaller.

mass loss rate is less than 1 %, so using silica sol as an inorganic porous carrier is feasible.

(2) DSC test results show that the start phase change temperature of PEG/SiO₂-CPCM is 42.4 °C. During the heating process, the theoretical enthalpy of PEG/SiO₂-CPCM is about 121.03 J/g, and the measured enthalpy is 82.1 J/g, the phase change enthalpy reduced by 38.93 J/g.

(3) With the increase of age, the strength of phase change concrete with different PEG/SiO₂-CPCM content decreases continuously, and the change of strength of phase change concrete of PEG-V5 group is the most obvious, the decline range gradually decreases from the 27.62 % of 3 d to only 4.96 % of 28 d, but the strength meets the requirement of C50.

(4) Under the premise of ensuring good working performance of the concrete, the addition of a certain amount of PEG/SiO₂-CPCM can greatly enhance the thermal conductivity of the concrete, but its thermal conductivity is reduced when it undergoes phase change under high temperature conditions.

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