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Effectiveness of the different eutectic phase-change materials in cooling asphalt pavement

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ABSTRACT

Choosing a phase-change material (PCM) adapted to the specific phase-change temperature (T_m) required for each temperature condition is of utmost importance in cooling pavements. Eutectic phase-change materials (EPCMs) realize the customization of the desired T_m and reduce the difficulty of matching PCMs. This work aims to investigate the effectiveness of a group of binary/ternary EPCMs with T_m ranging from 30 to 60 °C and melting enthalpies of around 200 J/g as thermal regulation components for different asphalt. To achieve this goal, the thermal and rheological properties of phase-change asphalt binders (PCAB) were evaluated by differential scanning calorimeter, thermogravimetric, Fourier transform infrared, and multiple stress creep and recovery tests. The results showed that PCAB with latent heat improved the specific heat capacity, which brought a maximum temperature lag of 134.5 min and a maximum temperature difference of 11 °C. Similarly, the PCAB remained chemical stability, and its thermal stability complied with the construction temperature specifications. However, the recrystallization of EPCMs was impeded by the molten asphalt binder matrix, resulting in a significant reduction in both the T_m and enthalpy. Therefore, these reductions should be taken into consideration when choosing an EPCM. Additionally, as liquid EPCM softens the binder, the absence of elasticity in solid EPCMs renders the binder stiff, thus reducing its resistance to deformation. These impacts were particularly noticeable in Styrene-Butadiene-Styrene modified asphalt due to the polymer network being dissolved by liquefaction-EPCMs. To sum up, EPCMs with a higher T_m (40–60 °C) may decrease their negative impact on deformation resistance, such as palmitic acid-myristic acid-methyl stearate mixtures.

1. Introduction

The characteristics of asphalt pavement are significantly affected by the prevailing climatic conditions, as mentioned in various studies [1,2]. The heat absorption coefficient is a widely used metric to describe a material's ability to absorb radiant energy, ranging from 0 to 1. A material with a high heat absorption coefficient of 1 indicates its capacity to fully absorb radiant energy, while a coefficient of 0 suggests no absorption [3]. The heat absorption capability of black asphalt pavement is quite impressive, with the potential to attain a heat absorption coefficient of 0.9. Under solar irradiation, the asphalt pavement exhibits efficient absorption and conversion of solar radiation into heat energy, leading to its progressive heating [4,5]. A graph, depicted in Fig. 1, exhibits temperature readings of asphalt pavement recorded in a test area located in Xi'an, Shaanxi Province, China, during the months of

July and August. It reveals that during the hot summer months, the asphalt pavement's temperature varies widely across its various layers, with a peak of 58 °C. Additionally, the temperature difference between daytime and nighttime was 20 °C. It has been reported in various studies that in hot regions, asphalt pavement temperature can rise as high as 70 °C when exposed to high levels of solar radiation [6,7]. Exposing asphalt pavements to high temperatures and the significant temperature difference result in various high-temperature distresses such as rutting and thermo-oxidative aging [8]. These distresses reduce asphalt pavement's functionality, safety, and durability [9]. In addition, asphalt pavement contributes to the urban heat island effect by generating significant amounts of heat and organic volatiles, leading to air pollution. This effect is further exacerbated by the properties of asphalt binder, which tends to absorb and retain heat, thereby increasing the surface temperature of the pavement [10,11]. Given the negative impacts of the

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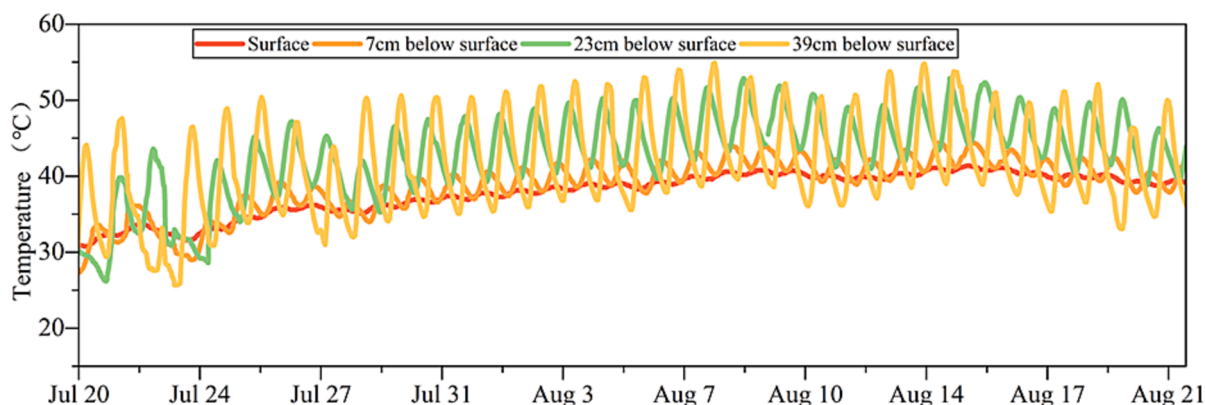


Fig. 1. The temperature of the test section of the asphalt pavement from July 20 to August 20.

environment on pavement performance, it is crucial to incorporate appropriate design elements that can mitigate these effects. These design elements can help counteract the detrimental effects of high temperatures, solar radiation, and other environmental factors, thereby improving the durability and overall performance of asphalt pavement.

To address pavement distress induced by high-temperature environments and mitigate the urban heat island effect, it may be feasible to depend on the latent heat characteristics of the phase-change materials (PCMs) to regulate the temperature field of the pavement [12,13]. A PCM must have a suitable phase transition temperature and high melting enthalpy to effectively be an active cooling agent for asphalt roads [14,15]. On the one hand, higher melting enthalpy is always accompanied by more excellent latent heat storage and release capacity [16]. On the other hand, when considering the application of PCM as a thermal regulation unit for cooling asphalt pavement, the phase-change temperature is critical in the material’s geographical applicability [17,18]. Many studies indicated that maintaining a phase-change temperature of 30–60 °C is necessary for incorporating PCMs into asphalt pavement [19]. For example, the shape-stable PCM made from paraffin was reported to have a melting temperature of 40–50 °C [20,21]. Wang et al., Zhang et al., and Gao et al. evaluated the temperature regulation effect of polyethylene glycol (PEG) with a melting temperature of 45–60 °C as a phase-change unit on the cooling pavement [22–24]. Jia et al. [25] investigated the potential of PEG of varying molecular weights as PCMs for cooling asphalt pavement. The palmitic acid-stearic acid combination was adsorbable by Jin et al. [26] using varying mineral

loadings, and its phase-change temperature was about 53 °C. Evidence from these analyses supports PCM’s use as a functional unit for regulating pavement temperature. However, the focus of applications has been primarily centered on PEG, paraffin (PW), and stearic acid (SA) [19]. Limited research on the effectiveness of asphalt pavements with varying phase transition temperatures is available.

On top of that, eutectic phase change materials (EPCMs) of organic fatty acids are a fascinating discovery among the PCMs under study, a combination of two or more components with different chemical and physical characteristics manufactured using a specific procedure to obtain the lowest possible melting point [27,28]. The eutectic method also realizes the customization of EPCMs of the desired melting point, dramatically reducing the difficulty of matching PCMs and broadening the phase-change temperature of PCMs with higher latent heat [29,30]. Only a small subset of EPCMs have been investigated, including adipic acid (CA), lauric acid (LA), picric acid (MA), palmitic acid (PA), and stearic acid (SA). In terms of melting point, Ahmet et al. successfully synthesized CA-PA [31], PA-MA [32], PA-LA [33], and MA-SA [34], with respective values of 21.85 °C, 42.6 °C, 35.2 °C, and 44.13 °C, and melting enthalpies of 171.22 J/g, 169.7 J/g, 166.3 J/g, and 182.4 J/g. CA-PA-SA/expanded graphite composites have a melting point of 21.33 °C and a latent heat of 131.7 J/g, according to the research conducted by Hua et al. [35]. Nan et al. [36] prepared LA-MA-PA/expanded graphite shape-stabilized composite PCM with a phase-change temperature of 30.94 °C and a latent heat of 135.9 J/g. A set of EPCMs based on fatty acids with 10–18 carbons, described by Ke et al. [37] and Nazir et al. [38], with phase-change temperatures between 15 °C and 50 °C. EPCMs have demonstrated successful application in thermal energy storage, while their utilization in asphalt pavement research remains limited. Meanwhile, more research efforts have been devoted to PCM’s packaging technique when introducing PCM as thermal regulation units for cooling asphalt pavement. To account for the significant temperature difference between different layers of asphalt pavements and the varying climate zones with distinct temperature conditions, it is crucial to carefully select PCMs that can adapt to the specific phase-change temperature required for each condition.

Therefore, it is necessary to investigate the effectiveness of PCMs with different phase-change temperatures in asphalt binders. A set of eutectic PCMs with varying phase-change temperatures (the interval of about 5 °C) and nearly identical phase-change enthalpy is derived through the eutectic method. Notably, due to the distinct high-temperature stability of different asphalt binders, it is imperative to conduct further investigations to ascertain the applicability of PCMs to each specific binder type. The objective of this study is to examine the feasibility of incorporating EPCMs with different phase-change temperatures in asphalt pavement. To achieve this, a range of binary/ternary EPCMs with phase-change temperatures ranging from 30 to 60 °C were prepared and added to three different types of asphalt at a rate

Table 1

Conventional indexes of 70 # base asphalt binder.

Properties	Values	Technical requirement	Specification
Penetration at 25 °C, 100 g, 5 s (0.1 mm)	71	60–80	JTG E20-2011 T0604
Penetration Index	−1.1	−1.5~+1.0	JTG E20-2011 T0604
Dynamic viscosity at 60 °C (Pa.s)	218	≥180	JTG E20-2011 T0620
Softening point (°C)	48	≥43	JTG E20-2011 T0606
Ductility at 10 °C (cm)	39	>20	JTG E20-2011 T0605
Ductility at 15 °C (cm)	>150	>100	JTG E20-2011 T0605
Wax content (%)	1.6	<2.2	JTG E20-2011 T0615
Flashpoint (°C)	297	≥260	JTG E20-2011 T0611
Density (g/cm ³)	1.033	–	JTG E20-2011 T0603
Solubility (trichloroethylene) (%)	99.95	≥99.5	JTG E20-2011 T0607

Table 2
Conventional indexes of 90# base asphalt binder.

Properties	Values	Technical requirement	Specification
Penetration at 25 °C, 100 g, 5 s (0.1 mm)	85	80–100	JTG E20-2011 T0604
Penetration Index	−1.4	−1.5~+1.0	JTG E20-2011 T0604
Dynamic viscosity at 60 °C (Pa.s)	173	≥140	JTG E20-2011 T0620
Softening point (°C)	45.5	≥44	JTG E20-2011 T0606
Ductility at 10 °C (cm)	>100	>30	JTG E20-2011 T0605
Ductility at 15 °C (cm)	>150	>100	JTG E20-2011 T0605
Wax content (%)	0.9	<2.2	JTG E20-2011 T0615
Flashpoint (°C)	262	≥245	JTG E20-2011 T0611
Density (g/cm ³)	1.032	–	JTG E20-2011 T0603
Solubility (trichloroethylene) (%)	99.92	≥99.5	JTG E20-2011 T0607

Table 3
Conventional indexes of SBS-modified asphalt binder.

Properties	Values	Technical requirement	Specification
Penetration at 25 °C, 100 g, 5 s (0.1 mm)	55	40–60	JTG E20-2011 T0604
Penetration Index	0.1	≥0	JTG E20-2011 T0604
Ductility at 5 °C (cm)	28	>20	JTG E20-2011 T0605
Viscosity at 135 °C (Pa.s)	2.3	<3	JTG E20-2011 T0625
Softening point (°C)	79	≥60	JTG E20-2011 T0606
Flashpoint (°C)	296	≥230	JTG E20-2011 T0611
Elastic recovery at 25 °C (%)	86	≥75	JTG E20-2011 T0662
Segregate, Soften Spreads (°C)	1.1	<2.5	JTG E20-2011 T0661

of 15%. The impact of phase-change temperature on the thermal properties, chemical structure, and rheological properties of the asphalt binders was analyzed. The effectiveness of temperature control was also assessed, and the results were summarized to assist engineers and researchers in selecting appropriate PCMs for use in cooling asphalt pavements.

2. Materials and test methods

2.1. Raw materials

In this study, three different asphalt binders were utilized, including the 70 # base asphalt binder (with 60/80 penetration), 90 # base asphalt binder (with 80/100 penetration), and

Table 4
Fundamentals of PCMs.

Item	CAS No	Linear Formula	Molecular Weight	Purity (%)	Phase-change temperature (melting, °C)	Phase-change enthalpy (melting, °C)
MS	112–61-8	CH ₃ (CH ₂) ₁₆ CO ₂ CH ₃	298.5	97	35.96	208.45
MA	544–63-8	CH ₃ (CH ₂) ₁₂ COOH	228.37	98	52.83	204.73
PA	57–10-3	CH ₃ (CH ₂) ₁₄ COOH	256.42	99	61.10	213.29
SA	57–11-4	CH ₃ (CH ₂) ₁₆ COOH	284.48	98	67.70	223.75
Butyramide	541–35-5	CH ₃ CH ₂ CH ₂ CONH ₂	87.12	98	115.42	229.49

styrene–butadiene–styrene (SBS) modified asphalt binder. The 70 # and 90 # base asphalt binders were sourced from Shandong Haiyun Asphalt Co., Ltd., and their properties are listed in Table 1 to Table 2.

The SBS-modified asphalt binder used in this study was obtained from Shandong Lutong Road Material Co., Ltd. in China, and its properties indices are listed in Table 3. The content of SBS in the modified asphalt binder is 4.2%.

The PCMs used in this study were Methyl stearate (MS), myristic acid (MA), palmitic acid (PA), stearic acid (SA), and butyramide, all of which were supplied by Shanghai Macklin Biochemical Technology Co., Ltd. The supplier provided the primary information, and the phase change temperature and enthalpy were established by a differential scanning calorimeter (DSC) in the laboratory. Some of their properties are presented in Table 4.

2.2. Determination of the mass ratio of binary/ternary eutectic PCMs

In a eutectic mixture, only one phase-change point, the lowest point in the diagram, corresponds to the solid–liquid phase-change. The melting point of the blended combination is always lower than the melting point of a single component [38]. To obtain PCMs with different phase-change temperatures, two or three organic fatty acids are mixed based on their eutectic point to create binary or ternary EPCMs. Equations (1) to (2) indicate that the theoretical molar ratios of a set of binary/ternary EPCMs were determined using the Schrader equation and then translated to theoretical mass ratios [39,40].

$$\ln X_i = \frac{\Delta H_i}{R} \left(\frac{1}{T_i} - \frac{1}{T} \right) \cdot (i = A, B) \quad (1)$$

$$X_A + X_B = 1 \quad (2)$$

where, X_i is the mole percentage of component i , T_i is the melting temperature of component i (K), R is the gas constant (8.314 J/(mol·K)), and ΔH_i is the molar heat of fusion of component i (J/mol).

The mass eutectic ratios of different binary EPCMs, such as butyramide-stearic acid (BSA, 0.154:0.846), butanamide-palmitic acid (BPA, 0.141:0.859), stearic acid-palmitic acid (SAPA, 0.398:0.602), butyramide-myristic acid (BMA, 0.125:0.875), palmitic acid-methyl stearate (PAMS, 0.276:0.724), and palmitic acid-myristic acid (PAMA, 0.386:0.614) were computed using the fundamental information data of organic chemical components in Table 4 and Equations (1) and (2). It should be noted that when calculating the eutectic point for a ternary EPCM, the mass ratio of the binary EPCM is taken into account along with the third phase. Based on this, the mass ratios of SA, PA, and MA in the ternary eutectic combination SAPAMA were 0.201:0.304:0.495, while the mass ratios of PA, MA, and MS in PAMAMS were 0.150:0.238:0.613.

2.3. Preparation for binary/ternary eutectic phase-change materials

The EPCMs were prepared based on our previous research [41,42]. To begin, place the appropriate mass of organic material on an analytical scale with an accuracy of 0.001 g and into an oven preheated to 90 °C (130 °C for BSA, BPA, and BMA). Butanamide has a melting point of 115.42 °C; hence EPCMs containing it must be prepared at a temperature of 130 °C. Once the samples were melted, they were put in a

Table 5

The acronyms of different modified asphalt binders.

EPCMs	70# base asphalt binder	90# base asphalt binder	SBS-modified asphalt binder
PAMAMS	70-PAMAMS	90-PAMAMS	SBS -PAMAMS
PAMS	70-PAMS	90-PAMS	SBS-PAMS
SAPAMA	70-SAPAMA	90-SAPAMA	SBS -SAPAMA
BMA	70-BMA	90-BMA	SBS -BMA
SAPA	70-SAPA	90-SAPA	SBS -SAPA
BPA	70-BPA	90-BPA	SBS -BPA
BSA	70-BSA	90-BSA	SBS -BSA
	70-EPCMs	90-EPCMs	SBS -EPCMs

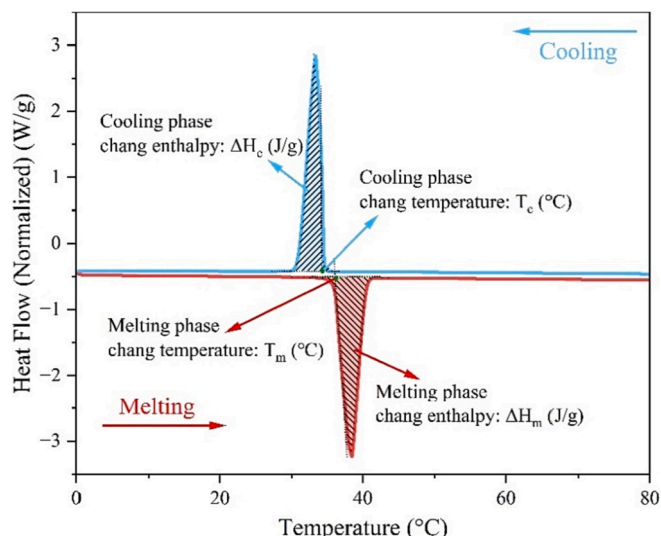


Fig. 3. Determination of the phase-change parameters.

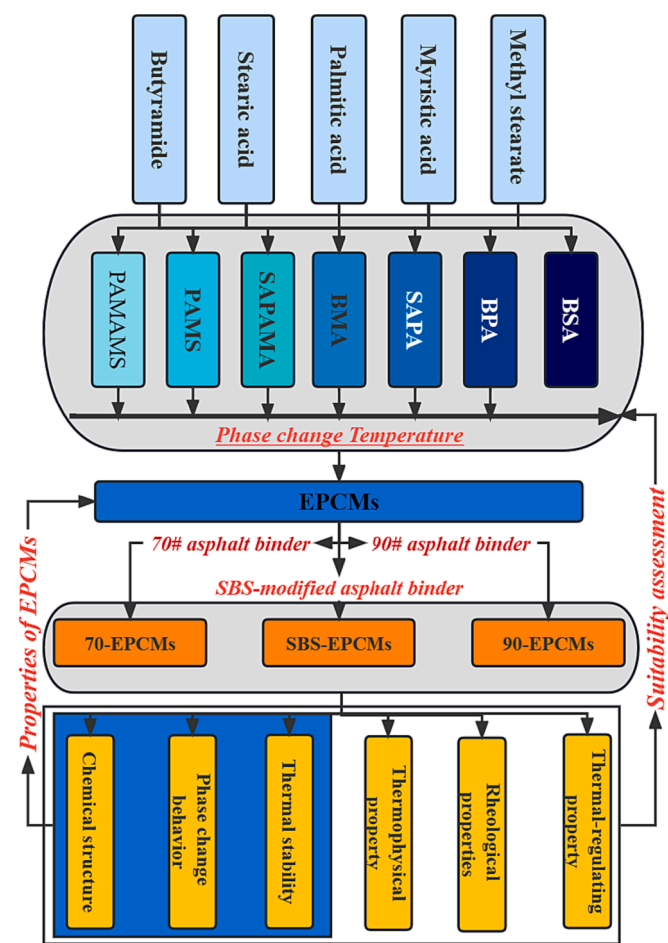


Fig. 2. Flowchart of this research.

magnetic stirrer and mixed at 300 r/min for 2 h to guarantee consistency. Finally, the liquid mixtures were cooled to obtain solidified EPCMs.

2.4. Preparation for phase-change asphalt binders

The high-speed shearing machine was applied to make the phase-change asphalt binder (PCAB). Since 15% SAPA content had been shown to have little effect on the asphalt binder while exhibiting outstanding temperature regulation performance in our earlier research [41,42], all EPCMs were added at that concentration (by mass of original asphalt binder). Following the molten EPCMs and the warmed asphalt binder, the base and the SBS-modified asphalt binder were sheared at 140 °C and 160 °C at 3000 rpm for 60 min. Twenty-one samples were produced for this investigation, and their respective acronyms are shown in Table 5.

2.5. Experimental design

The objective of this research was to investigate the feasibility of utilizing phase-change materials having different phase-change temperatures for cooling asphalt pavement. The detailed flowchart of the research process is illustrated in Fig. 2.

2.6. Characterization

The Fourier Transform Infrared (FT-IR) Spectrometer is a powerful tool for identifying chemical functional groups inside substances [43]. The reflection pattern of the Attenuated Total Reflection (ATR) of FTIR (NEXUS 870) was utilized to capture the spectra of EPCMs and PCAB to analyze their chemical structures. The spectrums were collected over 500 to 4000 cm⁻¹, with 32 scans performed for each measurement at 4 cm⁻¹. Three replicates of each sample were employed in the ATR-FTIR tests.

The phase-change behaviors were assessed using a DSC (DSC 250, TA Instruments, USA) with a heating/cooling rate of 5 °C/min and a flow rate of 50 ml/min of nitrogen atmosphere. The DSC device was calibrated using indium as a standard reference material, and the enthalpy measurement accuracy was 4%. Two heating-cooling cycles were conducted for each EPCM and PCAB, covering temperatures from -10 °C to 100 °C. Data from the second cycle was used for analysis. Furthermore, a 5-minute equilibration period was added after each heating/cooling cycle to eliminate thermal history and reduce measurement errors. Additionally, three replicate tests were performed on both EPCMs and asphalt binder samples. The software (TRIOS) was used to calculate thermal parameters such as melting enthalpy (ΔH_m), melting phase-change temperature (T_m), cooling enthalpy (ΔH_c), and cooling phase-change temperature (T_c), as shown in Fig. 3.

Thermophysical characteristics are crucial for temperature regulation when EPCMs regulate asphalt binder temperatures. The specific heat capacity is a fundamental parameter used to describe the thermo-physical characteristics of binders [44]. It refers to the amount of heat absorbed or released when the temperature of one unit mass of material changes by one degree Celsius [45]. The specific heat capacity of both EPCMs and PCAB was determined using the modulated differential scanning calorimetry (MDSC) mode. In this method, the sample oscillates at a specified frequency within a narrow temperature range. The specific heat capacity is then calculated by dividing the amplitude of the resulting oscillating heat flow by the amplitude of the oscillating heating rate. The MDSC test was performed using a temperature modulation

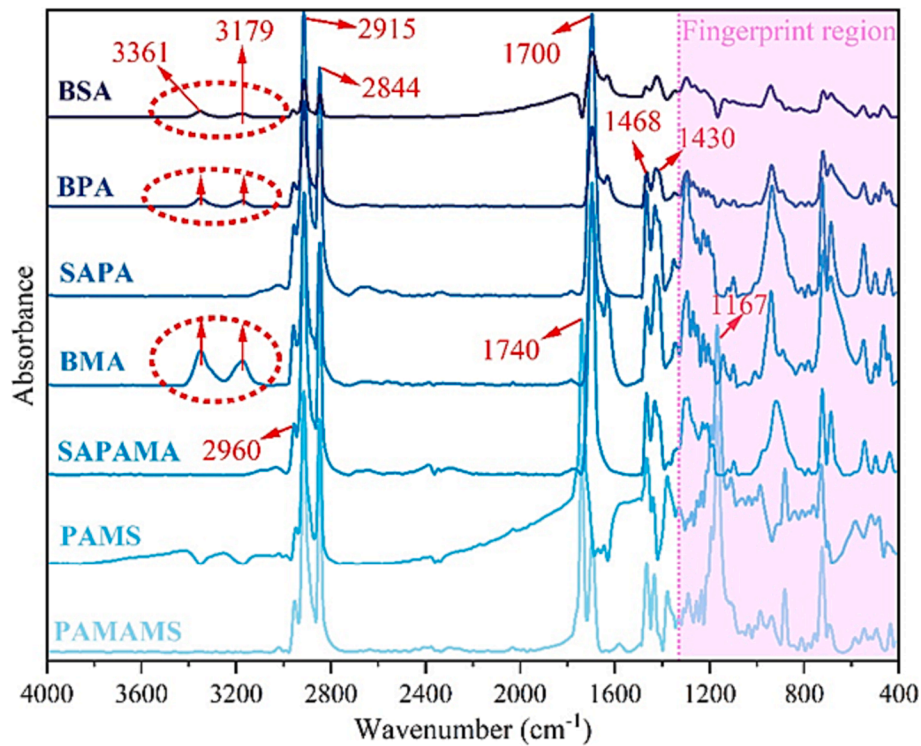


Fig. 4. FT-IR spectrums for EPCMs.

with a period of 100 s and an amplitude of 0.531 °C. The test was carried out under a nitrogen atmosphere with a flow rate of 50 ml/min, and the temperature was varied from 0 °C to 100 °C at a heating rate of 2 °C/min. The specific heat capacity was calibrated using sapphire. To obtain the “Reversible heat capacity (normalized) (J/g C)” signal, the test was replicated three times.

Pavement temperature regulation using PCM may keep roads within the range of temperatures where asphalt runs adequately, enhancing road performance, extending service life, and mitigating the urban heat island effect. Therefore, it is essential to investigate the temperature-regulating ability of PCAB, and the temperature–time curve is a straightforward way to characterize the temperature-regulating property. The temperature–time curves were obtained while exposing the sample to a constant heat source of 60 °C. To ensure that all samples had the same initial temperature, 25 g of the sample was placed in a beaker and kept in a thermostat at 15 °C for two hours before the test. The sample was placed in an environment box, and the heat source was turned on. The temperature rise was monitored and recorded using a PT100 temperature sensor and data recorder device. Two replicates were taken in the temperature–time curve tests.

To evaluate the thermal stability of EPCMs and PCAB, thermogravimetric analysis (TGA 2000, TA Instruments, USA) was utilized. Approximately 15 mg of each substance was heated at 10 °C/min in a nitrogen atmosphere until the temperature reached 600 °C. The corresponding DTG curves were determined to analyze the rate of thermal mass loss during the heating process. To ensure accuracy, the test was replicated three times.

The Multiple Stress Creep and Recovery (MSCR) is essential for evaluating the high-temperature rheological behavior of asphalt binders conducted by a dynamic shear rheometer (DSR, MCR 302, Anton Paar, Austria). The sample is loaded at constant stress for 1 s and then allowed to recover for 9 s. Twenty creep and recovery cycles are run at 0.1 kPa creep stress, followed by ten creep and recovery cycles at 3.2 kPa creep stress. Our previous research showed that the asphalt binder would lose its elasticity and viscosity after being introduced to liquefied PCM [42]. Since most EPCMs undergo a solid–liquid phase transition at about 28 °C

and 40 °C, these two temperatures were selected for the MSCR testing. Finally, the data from the 21st to 30th times were extracted to calculate the creep parameters, including average recovery percentage at 0.1 kPa ($R_{0.1}$), average recovery percentage at 3.2 kPa ($R_{3.2}$), average non-recoverable creep compliance at 0.1 kPa ($J_{nr0.1}$), average non-recoverable creep compliance at 3.2 kPa ($J_{nr3.2}$), according to the specification of ASTM D7405-2015. Two replicates were taken in the MSCR tests.

3. Results and discussion

3.1. Chemical structural analysis

It is widely accepted that the eutectic mixture is formed without any chemical reaction between the constituent components [32,49]. Fig. 4 displays the Fourier-transform infrared (FT-IR) spectra of EPCMs. The distinctive peaks observed in the spectra of EPCMs are attributed to the functional groups present in each component. Specifically, as illustrated in Fig. 4, the EPCMs contain high concentrations of saturated fatty acids (such as stearic acid, palmitate acid, and myristic acid) that possess similar molecular structures. This structural similarity is evident from the numerous strong peaks observed in the spectra of all samples at identical wavenumbers, including 2915 cm^{-1} , 2844 cm^{-1} , and 1700 cm^{-1} . These correspond to the antisymmetric stretching of the alkane CH_2 , the symmetric stretching of CH_3 , and the carboxylic acid carbonyl $\text{C}=\text{O}$ stretching of the carboxylic acid carbon group, respectively. Besides, the characteristic peaks at 1468 cm^{-1} and 1430 cm^{-1} represent the variable angle of the alkane CH_2 and the in-plane bending carboxylic acid COH . CH_3 antisymmetric stretching is responsible for the feeble absorption peak near 2960 cm^{-1} . Subsequently, some characteristic functional group absorption peaks appeared in different EPCMs. For instance, the NH_2 symmetrical stretching of butanamide may be seen in the spectra of BMA, BPA, and BSA, primarily at wavenumbers 3361 cm^{-1} and 3179 cm^{-1} . In the PAMAMS and PAMS spectra, the ester carbonyl group $\text{C}=\text{O}$ and the lactone $\text{C}-\text{O}-\text{C}$ in methyl stearate contribute strong characteristic peaks at 1740 cm^{-1} and 1167 cm^{-1} ,

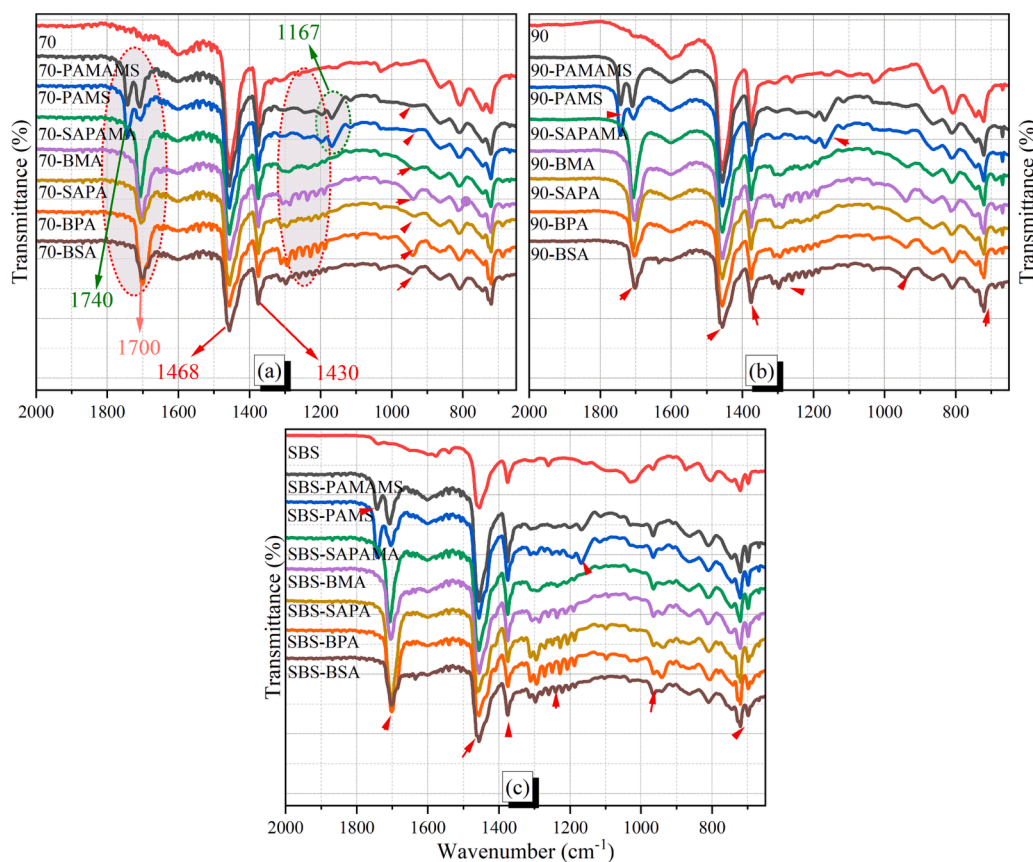


Fig. 5. FT-IR spectrums for asphalt binders: (a) 70-EPCMs, (b) 90-EPCMs, (c) SBS-EPCMs.

respectively. Likewise, the fingerprint region is defined as the range of wavenumbers between 1330 cm⁻¹ and 400 cm⁻¹. The vibration of the fingerprint region is the vibration of the whole molecule or a portion of the molecule and not the vibration frequency of a specific group [46]. The peaks around 1300–1200 cm⁻¹ and 950–900 cm⁻¹ are mainly attributed to the stretching and out-of-plane bending of carboxylic acid

C-OH. The C-C extension of the straight chain mostly corresponds to 1100–1020 cm⁻¹. The remaining peaks at lower wavenumbers (780–600 cm⁻¹) might result from the variable-angle vibrations of COO⁻. These conclusions also support the hypothesis that there is no chemical reaction during the co-crystallization of EPCMs, showing that the EPCMs have the same chemical properties as fatty acids. Sari et al.

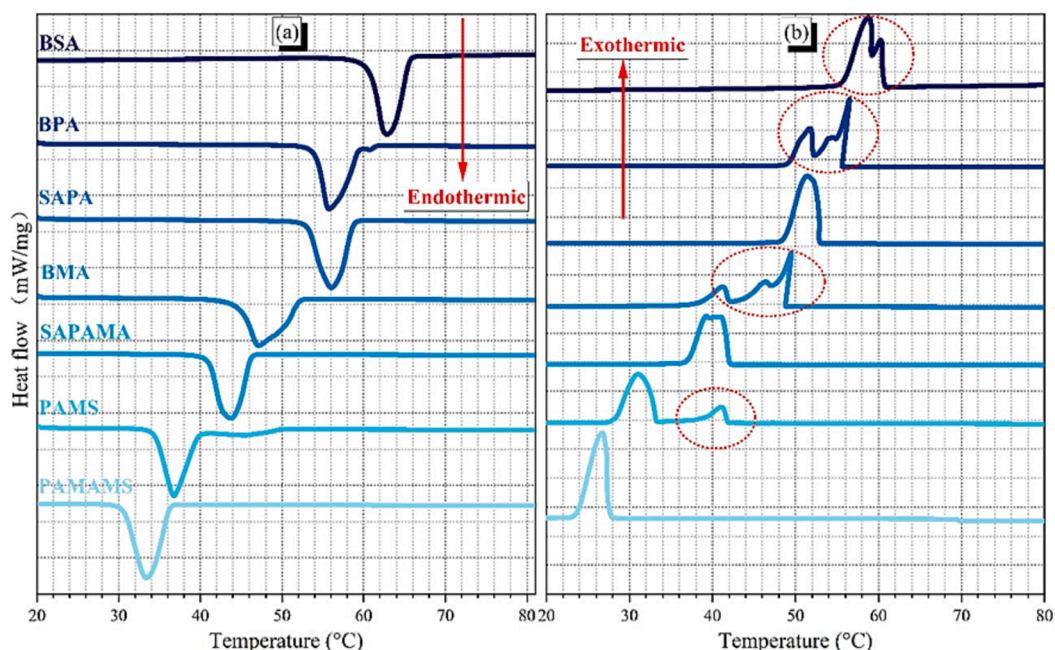


Fig. 6. Heat flow curves of EPCMs: (a) Endothermic curves, (b) Exothermic curves.

Table 6
Phase-change parameters of EPCMs.

EPCMs	ΔH_m (J/g)	T_m (°C)	T_m^i (°C)	ΔH_c (J/g)	T_c (°C)
PAMAMS	202.19	30.62	28.75	204.86	32.32
PAMS	210.06	34.45	33.65	212.46	33.25
SAPAMA	194.86	41.08	41.55	193.15	42.00
BMA	191.66	44.85	45.05	195.86	48.99
SAPA	201.13	53.12	53.45	204.14	52.82
BPA	197.90	54.17	55.65	192.14	55.81
BSA	200.34	60.80	60.85	209.15	60.50

[32] and Zhang et al. [47] reported the same conclusion in myristic-palmitic acid and palmitic-stearic acid eutectic mixtures, respectively.

The spectral structure of PCAB is altered upon the incorporation of EPCMs, as is evident from the spectra of the different asphalt binders shown in Fig. 5. Consensus among most sources is that only two strong characteristic peaks are observed at high wavenumbers in the spectra of binders; these peaks correspond to the antisymmetric stretching of the alkane CH_2 (at 2915 cm^{-1}) and the symmetric stretching of CH_3 (at 2844 cm^{-1}) [48]. Therefore, Fig. 5 only displayed the spectra with wavenumbers between 2000 cm^{-1} and 650 cm^{-1} to better deliver the spectral information at the low wavenumber. On the one hand, the peaks in the spectra of PCAB are identical to those of the original asphalt binders; they include the aromatic ring $C=C$ expansion (1600 cm^{-1}), the variable angle of the alkane CH_2 (1468 cm^{-1}), the in-plane bending carboxylic acid COH (1430 cm^{-1}), etc. On the other hand, while the spectra of PCAB have unique absorption peaks, they may correlate to those of EPCMs. Specifically, the ester carbonyl $C=O$ and the lactone $C-O-C$ in methyl stearate were responsible for the absorption peaks at 1740 cm^{-1} and 1167 cm^{-1} , respectively. Carboxylic acid carbonyl $C=O$ stretching and carboxylic acid $C-OH$ stretching account for the absorption peaks at 1700 cm^{-1} and $1300\text{--}1200\text{ cm}^{-1}$. Based on the infrared spectra analysis, it can be concluded that the addition of EPCMs does not significantly alter the chemical structure of asphalt binders. The spectrum of the PCAB is merely a superposition of the spectra of EPCMs and the original asphalt binders, with no new absorption peaks being observed, consistent with the results reported in most studies [25,41,49,50].

3.2. Thermal-regulating properties analysis

3.2.1. Phase-change behaviors

Two of the most critical factors in determining the effectiveness of PCMs in asphalt are the phase-change enthalpy and the phase-change temperature at which the materials undergo energy storage. Fig. 6 illustrates that the heat flow curves of EPCMs exhibited several endothermic and exothermic peaks at different positions, providing evidence that these materials can store and release heat. The endothermic and exothermic peaks of EPCMs showed a pattern of shifting to the right from PAMAMS to BSA, indicating a steady increase in their phase-change temperature. It is worth noting that specific samples, such as PAMS, BMA, BPA, and BSA, exhibited two peaks in the exothermic phase compared to the endothermic peak, which was attributed to phase separation phenomena that occurred during the crystallization process [51]. Table 6 lists the phase-change parameters of EPCMs during both endothermic and exothermic processes. During the endothermic process, the highest and lowest values of ΔH_m for EPCMs were 210.06 J/g and 191.66 J/g , respectively, with a 9.4 J/g difference. In general, the value of T_m increased from 30 °C to 60 °C with an interval of 5 °C . Meanwhile, compared with the experimental value, there are only 6.5%, 2.4%, 1.1%, 0.4%, 0.6%, 2.7%, and 0.08% differences in the theoretical value. These findings align with the selection criteria of this study. The difference between ΔH_m and the corresponding ΔH_c was relatively minor, with the most significant difference being observed in BMA, which was only around 4% (from 200.34 J/g to 209.15 J/g). The supercooling effect was amplified due to the phase separation that occurs during crystallization and causes a shift in T_c . However, the phase separation during the crystallization stage does not impact the temperature-regulating ability of EPCMs since their influence on pavement temperature is primarily exerted during the endothermic stage.

As shown in Fig. 7, the endothermic and exothermic phase-change parameters could be observed in the heat flow curves. The phase-change enthalpy during melting and crystallization was detected in all PCABs, indicating their varying temperature regulation capabilities. However, previous research has shown that when EPCMs are added to the asphalt binder, the binder matrix can hinder the recrystallization process of EPCMs, resulting in a significant decrease in both phase-

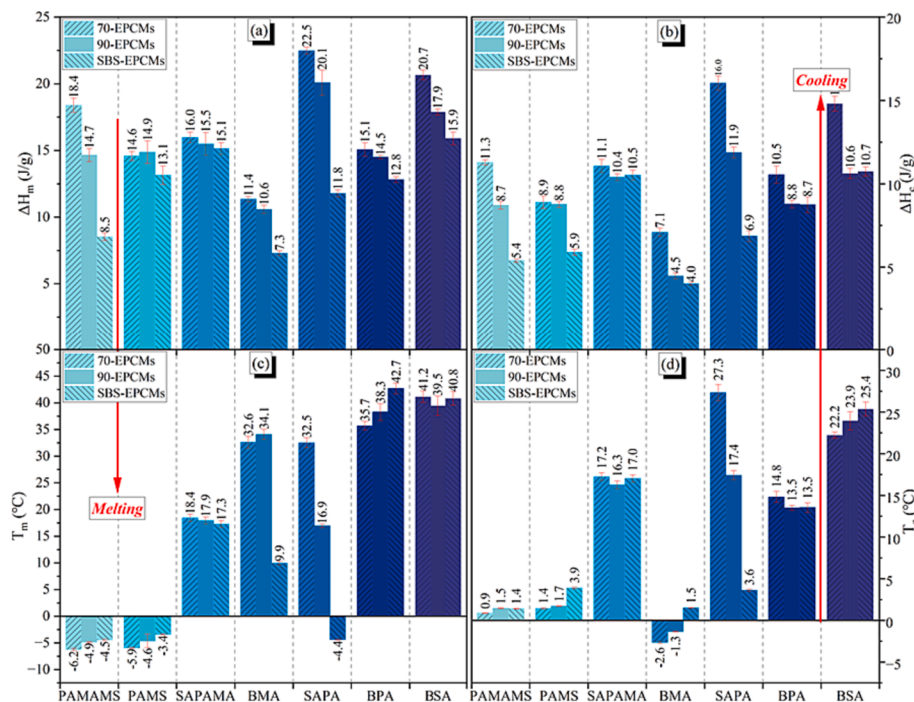


Fig. 7. The phase-change parameters of asphalt binders: (a) ΔH_m , (b) ΔH_c , (c) T_m , (d) T_c .

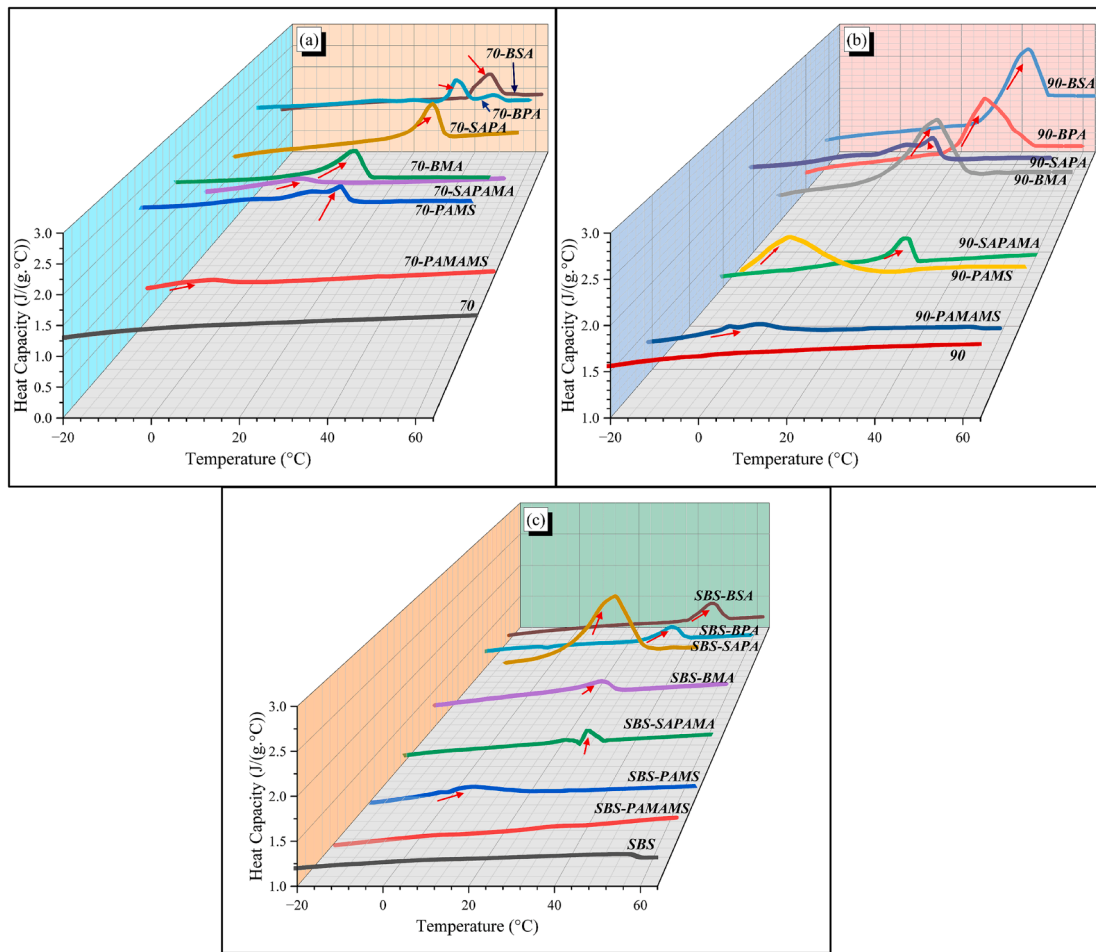


Fig. 8. Heat capacity curves for asphalt binders: (a) 70-EPCMs, (b) 90-EPCMs, (c) SBS-EPCMs.

change enthalpy (ΔH_m and ΔH_c) and phase-change temperature (T_m and T_c) [41,42]. It is also noteworthy that incorporating EPCMs into various asphalt binders resulted in a gradually declining trend of the ΔH_m and ΔH_c values of the corresponding PCMB. For instance, compared with 70-PCAB, the ΔH_m values of the corresponding SBS-PCAM decreased by approximately 52%, 40%, 35%, 61%, 47%, 42%, and 49% from PAMAMS to BSA, respectively. A similar trend could also be observed during the exothermic process. This result was attributed to the fact that the polymer network structure of SBS limits the migration of molecules and hinders the crystallization and melting of PCM, significantly reducing its phase-change enthalpy [25]. Furthermore, it is essential to note that the T_m value of the PCAB containing PAMAMS and PAMS was below 0 °C. This can be explained by two factors: first, the latent heat of PCAB is derived from PCM since there is no heat storage in the original asphalt binder; second, PCMs become liquid after melting, and the T_m of EPCMs is generally above 0 °C. Our previous studies have shown that liquefied PCMs would cause the binder to lose all elasticity and viscosity (the phase angle is almost 90°) [42]. PCMs undergo a continuous cycle of melting and crystallization. Although PCM can experience a complete melting and recrystallization cycle in the asphalt binder, its low crystallization temperature requires a cooler environment for the crystallization process to occur.

In summary, EPCMs as a temperature regulator in asphalt binder are most effective when the EPCMs undergo a complete melting and crystallization cycle at a higher phase transition temperature. Due to the low crystallization temperature of EPCMs, they may not be able to complete the crystallization process in most areas, resulting in the EPCMs always existing in a liquid state and losing their temperature-regulating effect in

the asphalt. Therefore, the latent heat function can be maximized by achieving a higher phase transition temperature when utilizing EPCMs as a temperature regulator for asphalt binders.

3.2.2. Specific heat capacity parameters

Fig. 8 shows the heat capacity curves of the PCAB obtained from the MDSC tests. The specific heat capacity is universally believed to reflect the atomic vibrational heat energy at the molecular level. EPCMs led to a rise in the specific heat capacity of asphalt binders, and some peaks corresponding to the heat flow curves were observed. These findings are partly explained by the fact that the latent heat of EPCMs causes heat accumulation, resulting in more energy required to increase the temperature of the binder [45]. Increasing the temperature induces a higher molecular movement velocity, demanding more energy [42]. For this reason, the specific heat capacity curves of all the binders exhibited an overall rising trend with increasing temperature, which is consistent with the research of Wang et al. [52]. A more significant specific heat capacity peak always accompanied the PCAB with higher phase-change temperatures. However, specific heat capacity curves of 70-PAMAMS, 90-PAMAMS, and SBS-PAMAMS were consistent with the original asphalt with no peaks, indicating that their thermoregulation function is lost at lower phase-change temperatures. Lastly, the specific heat peak of SBS-PCAB was always less than that of the corresponding 70-PCAB and 90-PCAB because the polymer network structure of SBS-modified asphalt considerably inhibited the re-crystallization of EPCMs [23]. In conclusion, these phenomena reveal, from the standpoint of thermo-physical characteristics, that EPCMs used to cool pavement typically need a higher phase-change temperature to guarantee that they can

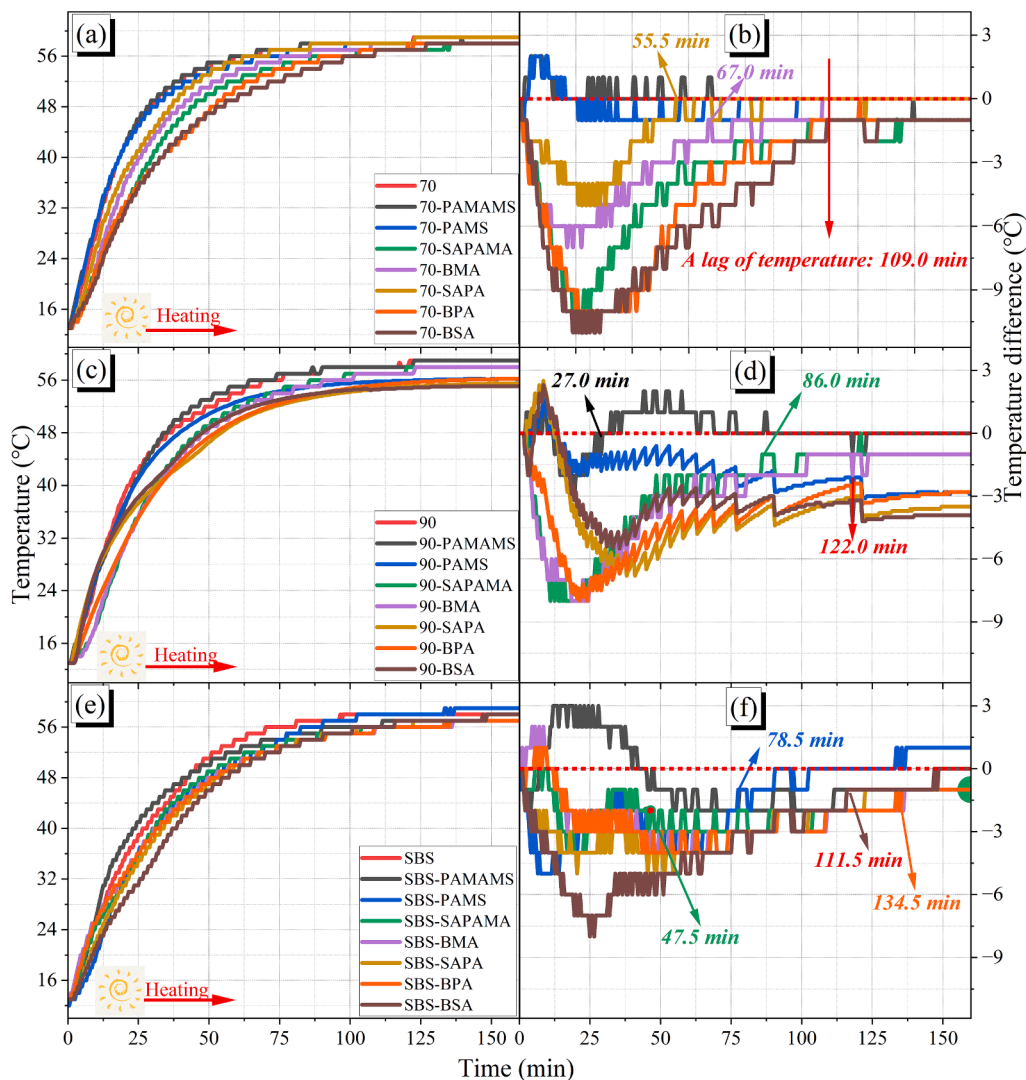


Fig. 9. Temperature-times curves: (a) (b) 70-EPCMs, (c) (d) 90-EPCMs, (e) (f) SBS70-EPCMs.

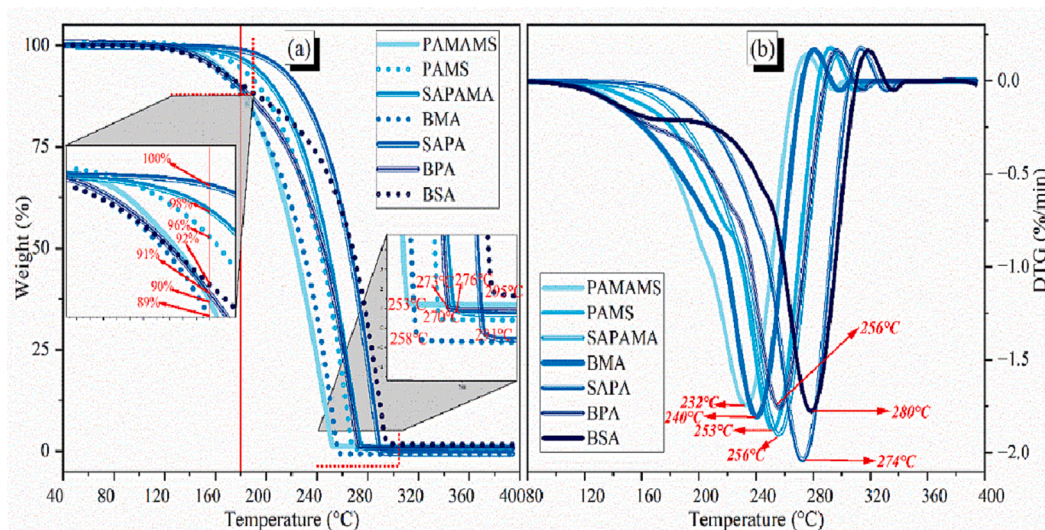


Fig. 10. TG and DTG curves of EPCMs: (a) TG, (b) DTG.

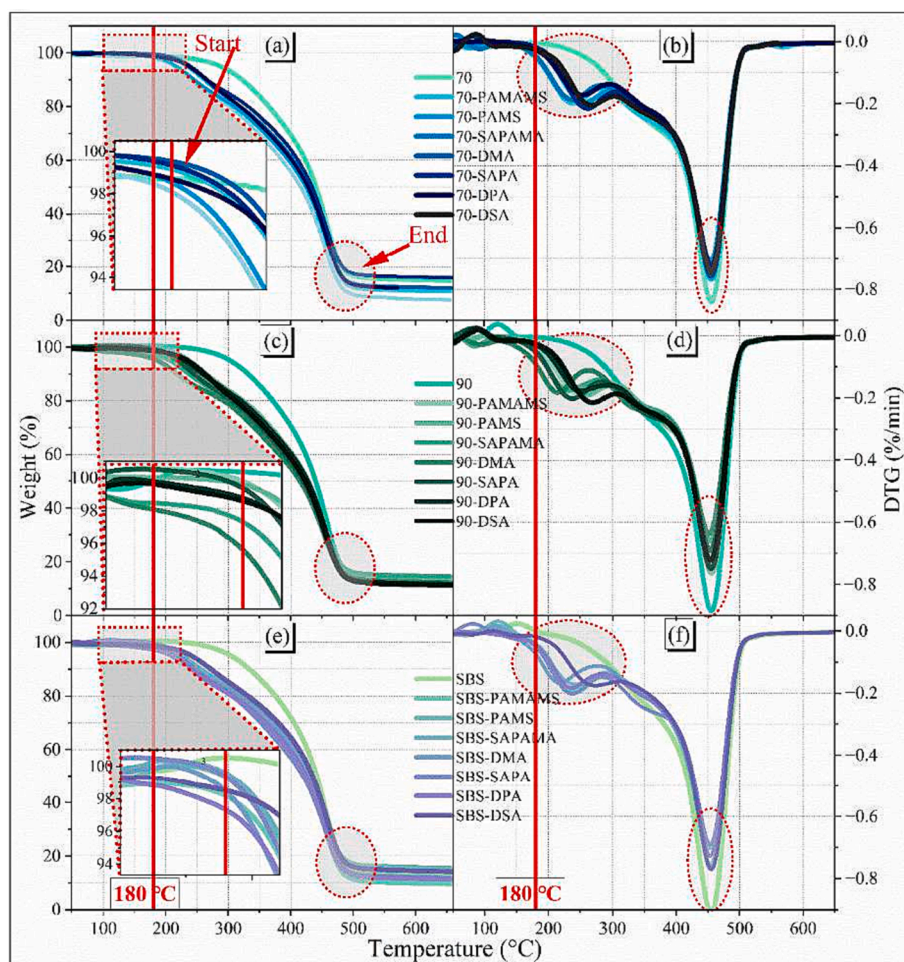


Fig. 11. TG and DTG curves for asphalt binders: (a) (b) 70-EPCMs, (c) (d) 90-EPCMs, (e) (f) SBS-EPCMs.

repeat the melting-recrystallization process at ambient temperature.

3.2.3. Temperature-time curves

As can be seen from Fig. 9 (a) to (f), when a constant heat environment (60 °C) was applied, the temperature of binders experienced a gradual increase, arriving and stabilizing at around 58 °C at different times. Different temperature differences (between the original asphalt) could be detected, which were caused by the thermal storage ability of EPCMs. In detail, the 70-BPA and 70-BSA PCABs had the most considerable temperature difference, at 11 °C, whereas the extreme temperature difference in other PCABs was also maintained at about 8 °C. The temperature lag refers to the difference in time for the binder temperature to stabilize during heating. Notably, there were no significant temperature lags or differences in 70-PAMAMS, 70-PAMS, and SBS-PAMAMS. The PCAB with the minimum temperature lag was 27.0 min for 90-PAMSMS, while the maximum was 134.5 min for SBS-BPA. Meanwhile, the temperature lag for other PCABs was approximately 100 min. The temperature lag means that the heating time is extended, and PCM is less susceptible to thermal shock and temperature degradation of asphalt binder, thereby improving its resistance to deformation [5,53]. Besides, due to the lower latent heat of SBS-PCAB, the impact of adding EPCMs to SBS-modified asphalt binder has a somewhat lower temperature-adjustment effect than that of base asphalt binder. Furthermore, it is also worth mentioning that adding PAMAMS to various original asphalt binders does not result in glaringly significant temperature differences but results in temperatures slightly higher than the originals.

Consequently, these observations point to EPCMs' function in

lowering the heat transfer efficiency and thermal perception of asphalt binders, which in turn causes a noticeable temperature difference and lag between PCAB and the original asphalt binder. On the one hand, the possible explanation is that EPCMs increase the specific heat capacity of asphalt binder, requiring more energy to maintain a constant temperature throughout the tempering process, resulting in a temperature difference. On the other hand, the EPCMs' latent heat contributes to the additional enlargement of the temperature difference, leading to the extraordinarily high value. Phase change enthalpy and specific heat capacity measurements corroborate this. Jia and Du reported a similar temperature difference when PEG was incorporated into asphalt [25,54], which verified the reliability of the results in this work. From the point of view of the temperature-adjusting property, this finding showed that the PCM with a lower phase-change temperature could not exert its latent heat function.

3.3. Thermal stability analysis

Fig. 10 illustrates the TG and (differential thermal gravimetric) DTG curves of EPCMs, which showed that these materials undergo three phases of thermal decomposition: early, rapid, and ultimate decomposition. To be suitable for temperature regulators in asphalt binder, the thermal pyrolysis temperature of PCMs must typically be lower than the construction temperature of hot mix asphalt mixture, which is usually less than 180 °C [55]. Based on their TG curves, it could be observed that EPCMs experienced a mild thermal degradation reaction at around 180 °C, with SAPA, SAPAMA, and PAMS losing less than 5% of their mass, while the other four EPCMs lost approximately 10%. In addition, the

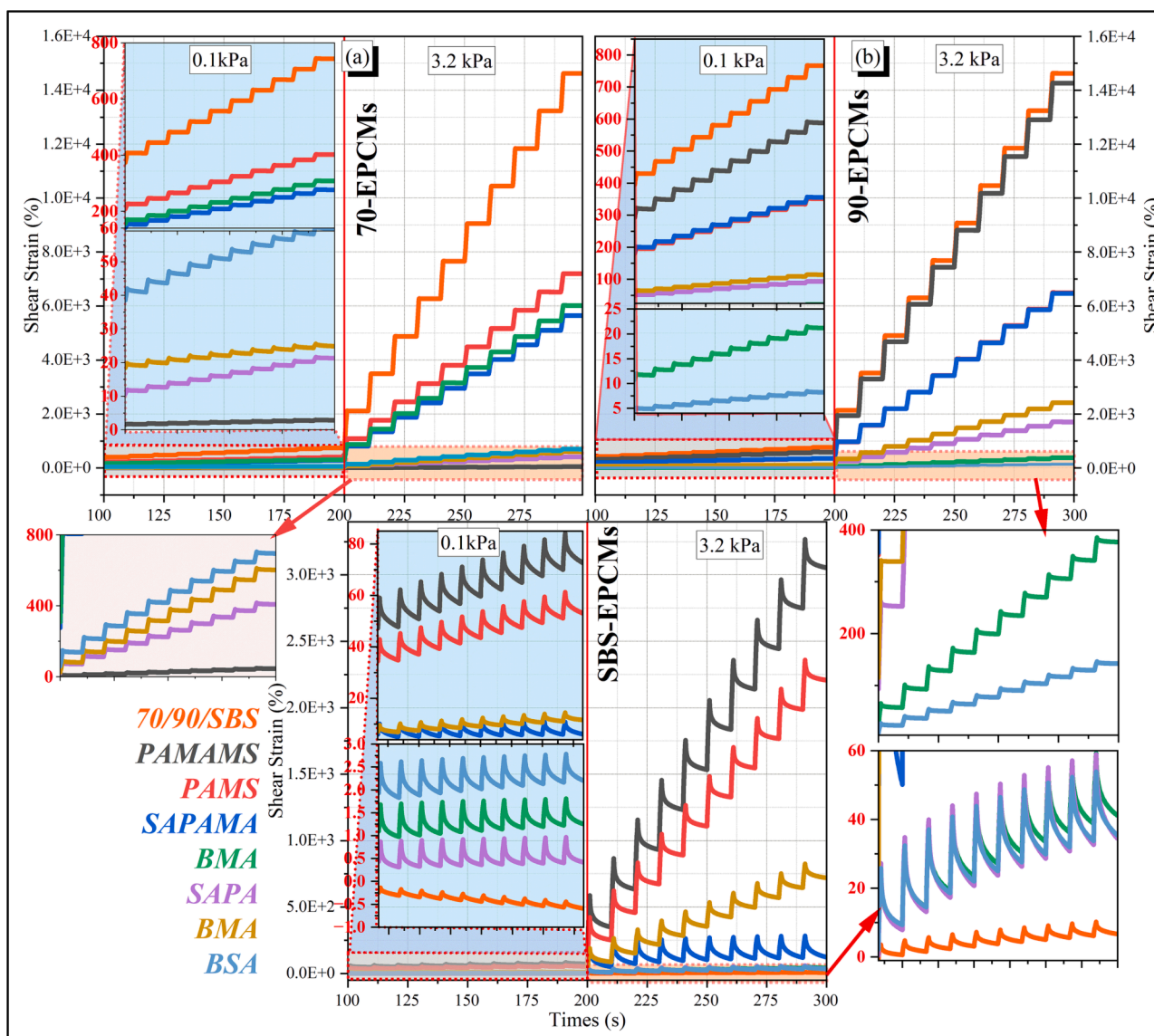


Fig. 12. The strain curves of asphalt binders: (a) 70-EPCMs, (b) 90-EPCMs, (c) SBS-EPCMs.

results indicated that SAPA, SAPAMA, and PAMS were more stable at high temperatures than the other EPCMs. Moreover, the DTG curves were also informative as they showed a parabolic shape for all EPCMs, with the maximum weight loss rate occurring at temperatures between 230 °C and 280 °C, well above the minimum temperature required for asphalt pavement construction. Finally, the EPCMs exhibited total thermal decomposition with a 100% mass loss occurring between 253°C, 270 °C, 276 °C, 258 °C, 291°C, 273 °C, and 295 °C. These results indicated that EPCMs effectively controlled the temperature of asphalt pavement while maintaining thermal stability.

The TG and DTG curves of all the PCAB exhibited a similar trend, as depicted in Fig. 11 (a) to (f). The initiation temperature of the thermal decomposition reaction for 70# base asphalt binder, 90# base asphalt binder, and SBS modified asphalt binder was approximately 200 °C, 223 °C, and 245 °C, respectively, while the final temperature of the thermal decomposition reaction was around 500 °C. In the initial decomposition stage, the TG curves of all PCAB were observed to be located below that of the original asphalt binders, indicating that the addition of EPCMs decreased the thermal stability of the asphalt. However, in the final decomposition stage, the mass loss rate of PCAB was lower than that of the original asphalt binder, suggesting that the incorporation of EPCMs improved the high-temperature stability of asphalt. In addition to the

volatilization of the light components of the binder itself, the thermal decomposition of PCAB was often followed by the decomposition of EPCMs, as seen by the TG curve of EPCMs. Conversely, PCAB lost less than 5% of its mass, and there was no noticeable thermal degradation reaction at 180 °C. Additionally, a prominent peak in the DTG curve of the original asphalt binders was observed, and its location, approximately 450 °C, was consistent with the other PCAB, which corresponds to the maximum mass loss rate of the asphalt binders. Two distinct peaks were observed comparing the DTG curves of PCAB samples to those of the original asphalt binders. The first minor peak, located around 220 °C, was caused by the degradation of EPCMs. The second peak, located at approximately 450 °C, was a prominent peak observed in the DTG curve of the original asphalt binders and is consistent with the other PCAB. Although the addition of EPCMs weakened the thermal stability of various asphalt binders, the resulting mixtures can still meet the temperature requirements for pavement construction.

3.4. Multiple stress creep and recovery analysis

The strain differential of PCAB as a function of time is affected by the strain level, the kind of EPCMs, and the type of original asphalt binder. Fig. 12 depicts the strain versus time at 28 °C, whereas Fig. 13 is a radar

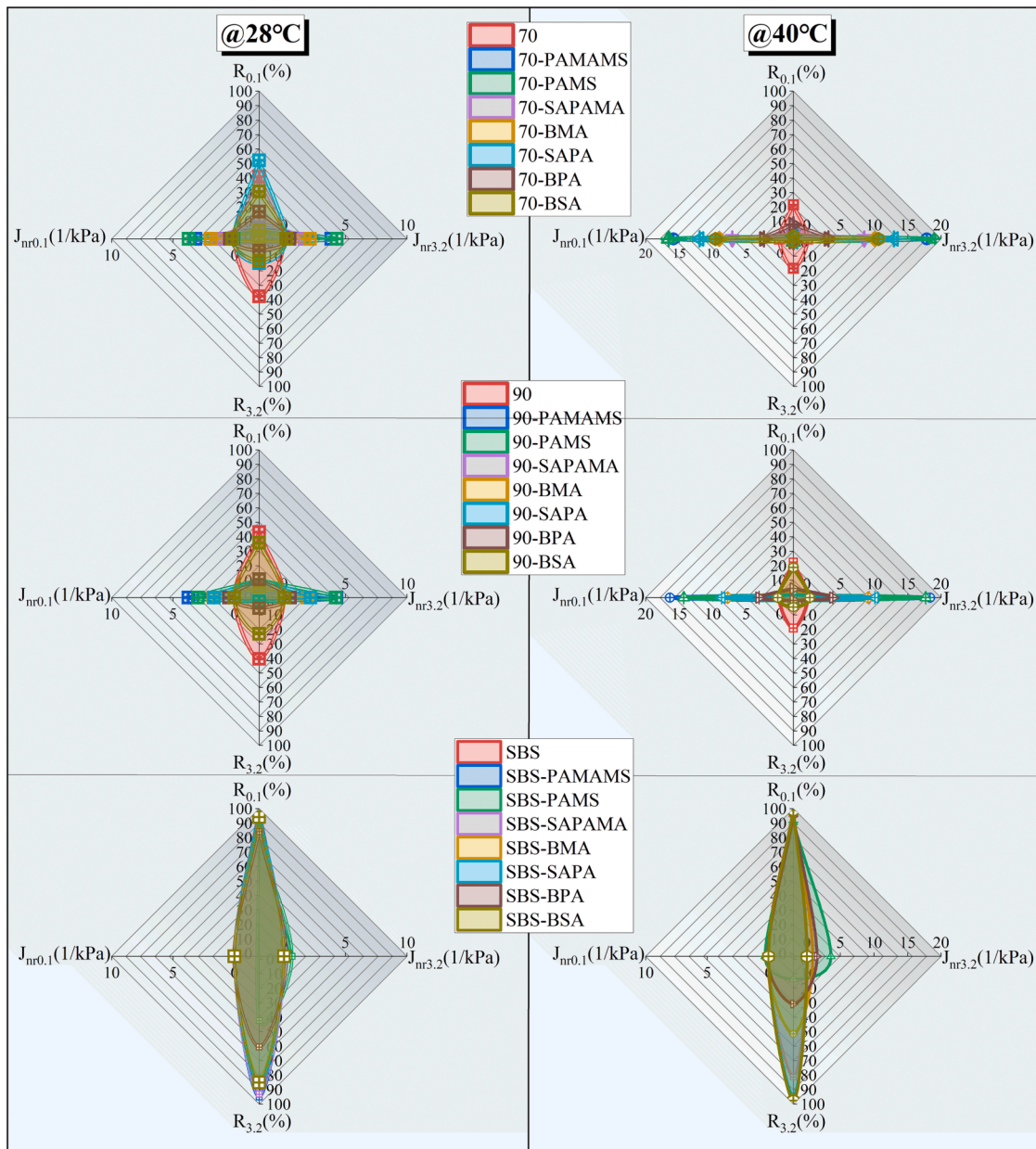


Fig. 13. The MSCR results of asphalt binders at 28 °C and 40 °C.

chart showing four crucial parameters (including $R_{3.2}$, $R_{0.1}$, $J_{nr3.2}$, and $J_{nr0.1}$) at 28 °C and 40 °C. The strain–time curves showed that the strain accumulation in PCAB was consistently more remarkable than that of the original asphalt binder under two stress levels. For instance, the cumulative strain of 70-PAMAMS, 90-PAMAMS, and SBS-PAMAMS at 0.1 kPa was about 258 times, 36 times, and 73 times that of the corresponding original asphalt binder, respectively. More enormous cumulative strains are often accompanied by poorer resistance to permanent deformation, indicating that EPCMs weaken the ability of asphalt binders to resist high-temperature deformation at the same stress level [56]. Nevertheless, the cumulative strain of 70-SAPA, 90-SAPAMA, and SBS-SAPA at 3.2 kPa was about 8.8 times, 2.7 times, and 5.2 times that of the original asphalt binder, respectively. The radar charts detected the most significant recovery for 70-SAPA, 90-SAPA, and 70-BSA at 40 °C. Thus, PCAB with a high phase-change temperature may mitigate the impairment of EPCM. The radar charts for 70-PCAB and 90-PCAB expanded to recovery at 28 °C, indicating a remarkable ability for the asphalt to recover from creep deformation. Also, the creep recovery part

of 70-PCAB and 90-PCAB decreased while the no-recovery part expanded at 40 °C, which was caused by the liquefaction of EPCMs. These findings showed that EPCMs were insensitive to the base asphalt grade. Meanwhile, the radar charts of SBS-PCAB with the nearly overlapping evolution performed the remarkable recovery properties of SBS-PCAB, which is derived from the slight effect of EPCMs on the creep properties of SBS-modified asphalt. The large $J_{nr3.2}$ of SBS-PAMS and low $R_{3.2}$ of SBS-BPA at 40 °C exhibited poor creep recovery at high-stress levels.

To sum up, the overall deformation resistance of PCAB is lower than that of virgin asphalt both before and after the phase-change of EPCMs. In fact, EPCMs may use the latent heat of phase-change to regulate the temperature of asphalt binders, thereby improving their performance at high temperatures. However, there is a lack of elastic capacity in solid-phase EPCMs; hence they cannot provide elastic components in the binder, resulting in brittle asphalt binders. Liquid EPCMs, on the other hand, soften the binder and drastically reduce its high-temperature stability. Based on AASHTO M 332–14, the original asphalt binder

passed through extreme traffic “E” at 28 °C and 40 °C. PCAB corresponded to a poor carrying level. For instance, 70-PAMAMA, 70-PAMS, 90-PAMAMS, and 90-PAMS only passed through standard traffic “S” at 28 °C. Notably, incorporating SA/PA, BPA, and BSA into binders could pass through the extremely heavy traffic “E” at 28 °C and 40 °C, indicating EPCMs have less impact on their creep recovery capabilities than previously thought, even if the accumulated strain is higher. Thus, from a material-selection perspective, EPCMs with higher phase-change temperatures may alleviate their negative influence on the deformation resistance of asphalt binders. Zhang et al. [23], Liu et al. [57], and Jinxi et al. [58] confirmed that the zero-leakage packaging strategy could eliminate the negative impact of liquefaction-PCM on the deformation resistance of the asphalt binder.

4. Conclusions and future work

The primary objective of this study was to determine the optimal phase-change temperature of EPCMs for the thermal conditioning of asphalt pavement during summer. Based on the findings, the following are the main conclusions:

(1) The study successfully developed a range of binary/ternary EPCMs with a melting phase-change enthalpy of approximately 200 J/g and a phase-change temperature of 30 °C to 60 °C. EPCMs and PCAB exhibited remarkable thermal and chemical stability.

(2) EPCMs provided a temperature lag of 27.5 to 134.5 min for PCAB, with a temperature difference of 8 to 11 °C. This can be attributed to the increase in PCAB’s specific heat capacity and specific heat curve peak. EPCMs such as PAMAMS and PAMS with low phase-change temperature exhibited poor temperature control and low specific heat capacity.

(3) The phase-change temperature and enthalpy values of PCAB decreased due to the slowdown of the recrystallization process of EPCMs caused by the molten asphalt binder matrix. This phenomenon was more pronounced in SBS-polymer PCAB due to its network structure. These reductions should be taken into consideration when choosing an EPCM to meet the requirements of stricter ambient temperature.

(4) The deformation resistance of PCAB was lower than that of virgin asphalt both before and after the phase-change of EPCMs. This is due, in part, to the fact that liquid EPCMs soften the asphalt binder, resulting in a significant reduction in its high-temperature stability. In contrast, the absence of elastic capacity in solid-phase EPCMs makes the binder brittle.

To achieve excellent temperature-regulating performance without significant impact on asphalt binder properties, the phase-change temperature of EPCMs used as asphalt pavement temperature adjustment units should not fall below 40 °C. Based on our findings, PCMs can be judiciously chosen for diverse application environments. In the future, the appropriate encapsulation strategies are necessary to address the potential impact of PCM liquefaction on asphalt properties. Also, a comprehensive assessment of the heat storage capacity of asphalt mixtures with PCMs is required, along with evaluating other mechanical performances. Last but not least, it is essential to emphasize that the cooling effect, enhancement of high-temperature performance, and long-term durability of PCM in real-world applications within asphalt pavement engineering merit further investigation.

CRedit authorship contribution statement

Jiasheng Dai: Writing – original draft, Methodology, Investigation, Funding acquisition. **Feng Ma:** Supervision, Funding acquisition, Conceptualization. **Zhen Fu:** Data curation. **Jenny Liu:** Writing – review & editing. **Chen Li:** Data curation. **Yingjie Hou:** Resources, Investigation. **Hanli Wu:** Writing – review & editing.

Declaration of Competing Interest

The authors declare the following financial interests/personal

relationships which may be considered as potential competing interests: None.

Data availability

Data will be made available on request.

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