Compatibility and Mechanical Properties of Epoxy Resin Modified Asphalt Binders

Peiliang Cong¹⁺, Shanfa Chen², Jianying Yu³, and Huaxin Chen⁴

Abstract: Epoxy asphalt binder is an excellent road material. A good compatibility between the epoxy resin and asphalt fulfills the basic requirement to achieve the goal of improving asphalt performance. The compatibility, morphology and mechanical properties were analyzed and evaluated by combining hot storage test with soxhlet extraction method, fluorescent microscopy and mechanical test, respectively. The results indicated that the compatibility and the flexibility of epoxy asphalt are bad, and these properties can be improved by adding solubilizer and styrene-butadiene-styrene (SBS) triblock copolymer. The effects of solubilizer and SBS contents on the compatibility, mechanical properties and morphologies of the epoxy asphalt are investigated. The epoxy asphalts showed better compatibility and elongation at break when solubilizer and SBS were added. Therefore, the use of solubilizer and SBS can be considered a suitable alternative for modification of epoxy asphalt in pavement.

Key words: Compatibility; Elongation at break; Epoxy asphalt; SBS; Solubilizer.

Introduction

Asphalt is an old, low cost, thermoplastic material. Petroleum asphalt became available through petroleum refining about 100 years ago. Asphalt is made up of a mixture of aliphatic, aromatic and naphthenic hydrocarbons and small quantities of organic acids and heterocyclic components containing nitrogen, oxygen, sulphur and metal atoms. However, the chemical composition of asphalt cannot be defined exactly, because it is extremely complex and variable. Its makeup depends on the source of the crude oil from which the asphalt originates and on modification induced by treatments in the refinery or during the "in service" life [1-2]. Therefore, the components of asphalt are usually grouped into two categories: asphaltenes and maltenes. The latter is further subdivided into saturates: aromatics and resins.

For a long time, asphalt has been widely used as flexible pavement construction binder materials [3-4]. However, the increasing traffic volume and vehicle loads demand high performance pavement, particularly on orthotropic steel bridge deck. Compared with the common asphalt pavement, asphalt concrete on steel bridge deck should deform with the steel bridge deck very well and have better heat resistance [5-11]. Thus, the properties of conventional asphalt mixture can not be considered satisfactory. The

use of polymer modified asphalt to achieve better asphalt pavement performance has been observed for many years [11-16]. The styrene-butadiene-styrene (SBS) triblock copolymer, styrene butadiene rubber (SBR), ethylene vinyl acetate (EVA) copolymer and polyethylene (PE) are the most common asphalt modifiers. Still, this polymer modified asphalt always retains thermoplasticity, which means that the asphalt binder flows at higher temperature [17-23]. Therefore, the above mentioned polymer modified asphalt can not satisfy the rigorous demands of the steel deck bridge surface paving because of their thermoplastic natures.

Previous works of research have confirmed that the performance of asphalt can be improved dramatically by epoxy resin [3, 4, 6-11]. For example, it can decrease thermal susceptibility and permanent deformation, increase fatigue resistance and low temperature cracking resistance, provide superior substrate adhesion, and is 3-4 times stronger than conventional asphalt concrete. However, the dielectric constant of epoxy resin and asphalt is 3.9 and 2.6-3.0, respectively. The major restriction in epoxy resin modified asphalt is the incompatibility of epoxy resin and asphalt, because epoxy resin is a polarity material and asphalt is a non-polarity material. To achieve the goal of improving asphalt properties, basic requirements dictate that the selected modifier should be compatible with asphalt [24-25]. Obvious differences between asphalt and epoxy resin exist, such as molecular weight, density, viscosity, solubility coefficient and so on. All of those result in bad compatibility between epoxy resin and asphalt. Therefore, it is one of the basic requirements for epoxy asphalt that epoxy resin and asphalt not separate or settle before the epoxy asphalt cures. Low compatibility between epoxy resin and asphalt may result in poor stability, which in turn leads to separation of epoxy resin and asphalt phases and inconsistent binder quality. In addition, epoxy asphalt concrete should deform with the steel bridge deck [26-33]. Therefore, it is required that epoxy asphalt have better flexibility.

The data concerning epoxy asphalt has been scarce until now. We need to provide more studies to solve the incompatibility and ductility of epoxy resin and asphalt. The primary goal of the present study is to improve compatibility and elongation at the break of

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epoxy asphalt binders by solubilizer and SBS. In addition, the study investigates the effects of solubilizer and SBS content on the compatibility and elongation at break of epoxy asphalt.

Experimental

Raw Materials

The 60/80 pen grade asphalt was used, with a penetration of 78 dmm at 25°C, softening point of 45.9°C, and viscosity of 0.50 Pa.s at 135°C. The Yueyang Petrochemical Co., Ltd., in China produced grade 791 using SBS. It is a linear-like SBS, containing 30 wt% styrene, and the average molecular weight of SBS is 120,000 g/mol. Epoxy resin is diglycidyl ether of the bisphenol A type, and its epoxy value is 0.52 mol/100 g. It was made in Shanghai Xinhua Resin Co., Ltd. in Shanghai, China. Methl-tetrahydro phalic anhydride, the curing agent, was provided by Jiaxing Fine Chemical Co., Ltd in Zhejiang Province, China. The solubilizer was prepared in a laboratory, and it had surfactant properties and contained a hydrophobic and hydrophilic group.

Methods

The stoichiometric proportion of epoxy resin and anhydride was controlled at 10:7, and the solubilizer concentration was added according to the total of epoxy asphalt. The concentration of SBS in asphalt were 0 wt%, 1 wt%, 2 wt%, 3 wt% and 5 wt%, respectively. In an oil-bath heating container, asphalt was heated to 170±5°C until it flowed fully. The SBS was mixed into the asphalt under a high rotation speed of about 60 minutes to ensure that the blend became essentially homogenous. When the temperature of the SBS/asphalt blend was reduced to 120°C, the appropriate amount of solubilizer and curing agent were added to the blend and mixed for 30 minutes with a lab mixer set adequately fast (usually 500 rpm) to create a small vortex without whipping excessive air into the sample. The epoxy asphalt was obtained when the desired amount of epoxy resin was added into the blend, and the mixer continued to stir the blend for 5 minutes under the same mixing condition.

Fourier Transform Infrared (FTIR) Spectroscopy (NEXUS, Thermo Nicolet, USA) was used to obtain the IR spectra of epoxy asphalt. The sample was prepared by casting film onto a potassium bromide (KBr) thin plate, and the spectra were obtained by 4 cm⁻¹ resolution.

The compatibility of epoxy asphalt binders was tested in the following manner. The epoxy asphalt was poured into an aluminum foil tube 25 mm in diameter and 140 mm in height. After sealing the tube without air enclosure, it was stored vertically and immovably in an oven at 120±5°C for 8 hours. Then the tube containing the epoxy asphalt was left in the oven and allowed to cool gradually to an ambient temperature. The cylindrical asphalt sample was cut horizontally into three equal sections. The Soxhlet extraction method (ASTM C613M-97(2008)) was employed to extract the samples from the top and bottom in an aluminum foil tube. The solvent is toluene. The control test proved that toluene can dissolve asphalt, but it cannot dissolve cured epoxy resin. The leftover solid matter was dried in an oven at 60°C and weighed. The amount of leftover in the sample was calculated as follow:

% leftover =
$$\frac{m_1 - m_0}{m - m_0} \times 100$$
 (1)

where m =The weight of total solid containing filter paper before extraction.

 m_0 = The weight of filter paper,

 m_1 = The weight of total solid containing filter paper after extraction Fluorescent microscopy was used to study the morphology of epoxy asphalt by observing the nature of the continuous phase and the dispersion of the discontinuous phase. Fluorescent microscopy is based on the principle that when epoxy asphalt is illuminated by fluorescence, the asphalt-rich phase appears dark or black, and the epoxy resin-rich phase appears white or yellow. In this study, a reflection fluorescent microscope (Model YS100, Yuyangfeng Co., China) equipped with an electron vidicon and a blue filter system was used to observe the morphology of epoxy asphalt. Squashed slides of epoxy asphalts were prepared using very thin films of the sample and viewed under the microscope at a magnification of 400 at ambient temperature.

The tensile strength and elongation at break of epoxy asphalt were tested on the Electro-Hydraulic Servocontrolled Testing System (Model Instron 1341, Instro LTD. U.K), referring to ASTM D638 at 23°C. The strain rate was 500 mm/min.

Results and Discussion

FTIR test

Over the past few years, with the support of the asphalt industry, the state highway departments in some countries and the Strategic Highway Research Program (SHRP) have done in-depth investigations of the chemistry of asphalt materials. FTIR spectroscopy was used to study the distribution of functional group types present in the asphalts. IR spectroscopy proved to be a very useful technique in analyzing structure in the fractions of asphalts. Fig. 1 gives Infrared spectroscopy analysis curves of three kinds of asphalt binders at room temperature. The strong peaks within the 2850-2960 cm⁻¹ region are typical C-H stretching vibrations in aliphatic chains. The C-H asymmetric deforming in CH2 and CH3 and the C-H symmetric deforming in CH_3 vibrations are observed at 1400-1500 cm⁻¹ and 1370-1390 cm⁻¹. The characteristic absorption peak around 1545-1640 cm⁻¹ is attributed to C=C stretching vibrations in aromatics. Besides the above absorption peak, epoxy asphalt also shows a strong peak at 830 cm⁻¹. 1509 cm⁻¹ is due to p-phenylene groups, 906 cm⁻¹ is due to the epoxy group, and the peak at 1035 cm⁻¹ results from the aliphatic carbon-oxygen stretching (-O-CH₂-). The peak revealed around 1740 cm⁻¹ is ester carbonyl of the cured product of epoxy asphalt. The results indicated that three kinds of asphalt show different chemical compounds, especial epoxy asphalt.

Compatibility

Asphalt has very delicate balances of polar to non-polar, homogenous to heterogeneous, small compounds to large compounds, associating components to dispensing solubilizing components, and aromatic to paraffin components and metals. This

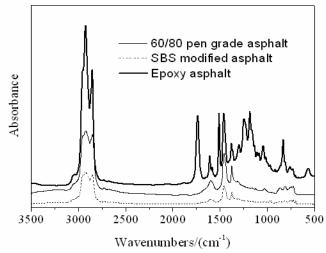


Fig. 1. Results of the IR Test of Asphalt Binders (SBS Modified Asphalt with 3 wt% SBS and Epoxy Asphalt with 50 wt% Epoxy Resin).

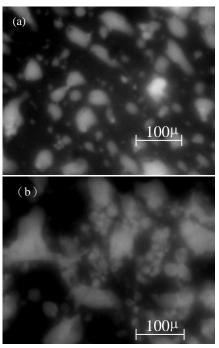


Fig. 2. Morphology of Control Epoxy Asphalt: (a) 30 wt% Epoxy Resin; (b) 50 wt% Epoxy Resin.

delicate balance of the mixture plays a key role during modification and the performance properties of asphalt and/or modified asphalt. Any imbalance in the chemistry of this delicate mixture results in incompatibility between asphalt and modifier. Due to the difference in the molecular weight, density, viscosity, solubility parameter and polarity between epoxy resin and asphalt, phase separation would take place in epoxy asphalt. Droplets of the epoxy resin dispersed in asphalt are usually accumulated and slinked down the asphalt during transportation. Epoxy asphalt is a thermodynamically immiscible mixture. When epoxy resin is blended with asphalt, epoxy resin is not soluble in asphalt, or asphalt is not soluble in epoxy resin. Epoxy resin was dispersed as large particles in the asphalt at the studied temperature and a magnification of 400, as Fig. 2 showed.

Table 1. Effect of Solubilizer on Compatibility.

	Epoxy Asphalt	Top/ (%)	Bottom $/(\%)$	Δ/(%)
	Control	32.2	54.4	22.2
0	.5wt% Solubilizer	33.3	52.9	19.6
1	.0wt% Solubilizer	34.5	49.5	15.0
1	.5wt% Solubilizer	34.9	47.6	12.7
2	.0wt% Solubilizer	35.6	46.1	10.5
2	.5 wt% Solubilizer	35.7	45.7	10.0

Table 2. Effect of SBS and Solubilizer on Compatibility.

Epoxy Asphalt	Top/(%)	Bottom/ (%)	$\Delta/(\%)$
Control	32.2	54.4	22.2
Solubilizer	35.6	46.1	10.5
1wt%SBS+ Solubilizer	36.7	45.5	8.8
2wt%SBS+Solubilizer	37.6	44.7	7.1
3wt%SBS+Solubilizer	38.0	44.2	6.2
5wt%SBS+Solubilizer	38.7	43.7	5.0

Note: Epoxy resin contents is 30 wt%, Solubilizer contents is 2 wt%.

The light phase in the picture represents the epoxy resin, and the dark phase is the asphalt. The results indicate that the compatibility of epoxy asphalt is bad, and the higher epoxy resin, the worse the compatibility.

These suspended epoxy resin particles in asphalt have descent tendencies, albeit gentle, under the influence of gravitational fields due to the density difference between epoxy resin and asphalt. It is adverse to achieving the goal of improving asphalt performance. The combination of storage test and Soxhlet extraction was employed to evaluate the compatibility of epoxy asphalt in this study. The solubilizer and SBS were adopted to improve the compatibility and elongation at break of epoxy asphalt, respectively. The effects of solubilizer and SBS on compatibility of epoxy asphalt are shown in Table 1 and Table 2.

Table 1 shows that the leftover gap between the top and bottom of the tube is large for control epoxy asphalt. However, the leftover gap between the top and bottom of the tube decreases when the contents of solubilizer increase. It becomes 10.0% when 2.5 wt% solubilizer is present in epoxy asphalt. The results indicate that the compatibility of epoxy asphalt was improved significantly when solubilizer was added to epoxy asphalt. The leftover gap between the top and bottom of the tube is 19.6%, 15.0%, 12.7% and 10.5% when 0.5 wt%, 1.0 wt%, 1.5 wt% and 2.0 wt% compatibility was added into epoxy asphalt, respectively. From 2.0 wt% to 2.5 wt%, there has been a little increment in leftover gap. Thus, 2.0 wt% solubilizer was selected to prepare the epoxy asphalt.

Table 2 demonstrates that the leftover gap between the top and bottom of the tube is 22.2% for control epoxy asphalt. It becomes 10.5% when solubilizer is present in epoxy asphalt. The results indicate that the compatibility of epoxy asphalt improves significantly when solubilizer is added into epoxy asphalt. When 1 wt%, 2 wt%, 3 wt% and 5 wt% SBS were blended with epoxy asphalt containing solubilizer, the leftover gap between the top and bottom of the tube is 8.8%, 7.1%, 6.2% and 5.0%, respectively.

The experimental results indicate that epoxy asphalt with better compatibility can be prepared successfully by adding solubilizer and

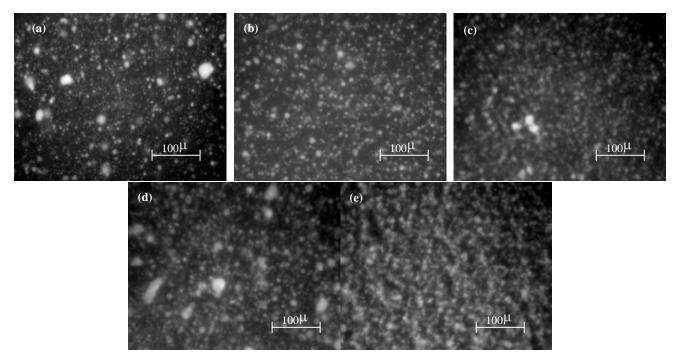


Fig. 3. Fluorescent Micrograph of Epoxy Asphalt: (a) Solubilizer, (b) Solubilizer + 1 wt% SBS, (c) Solubilizer + 2 wt% SBS, (d) Solubilizer + 3 wt% SBS, (e) Solubilizer + 5 wt% SBS.

SBS.

Morphology

The compatibility of epoxy asphalt containing solubilizer and different contents of SBS was studied using fluorescence microscopy. The method characterizes the nature of the continuous phase and the fineness of the dispersion of the discontinuous phase. The morphologies of epoxy asphalt are shown in Fig. 3. The light phase in the picture represents the SBS and epoxy resin, and the dark phase is the asphalt.

The morphology of epoxy asphalt containing solubilizer is remarkably different from that of control epoxy asphalt, as shown in Fig. 2. The blends of epoxy resin and asphalt exhibit a multiphase morphology. Epoxy asphalt shows the morphology of a continuous asphalt phase with dispersed small epoxy resin particles (Fig.3 (a)). Epoxy resin particles become fine when solubilizer are added into epoxy asphalt, and epoxy resin segregates into small regions, which is then known as the discrete phase. The images show a clear change in the morphology of the epoxy asphalt as the SBS content increases. Thus, SBS can alter the morphology of epoxy asphalt. The interlocked continuous phase forms with SBS content increasing. When the content of SBS is 2 wt%, the epoxy resin and asphalt are continuous and interlocked, as illustrated in Fig. 3(c). At this moment, the epoxy resin and SBS phase gradually become the matrix of the system, and the epoxy asphalt starts the phase inversion. This phase inversion results from the SBS content increase in epoxy asphalt. When the epoxy resin and SBS phase forms the continuous phase, as shown in Fig. 3(d), the properties of the mixture are mainly determined by the epoxy resin and SBS. Such systems generally pose higher cohesion and strength. The mechanical tests confirm the tensile strength of epoxy asphalt increase remarkably at low content epoxy. At a higher SBS content, a continuous epoxy resin and SBS phase with dispersed asphalt phase is observed, as shown in Fig. 3(e). The image demonstrates that the epoxy resin and SBS is in the continuous phase of the system, and the asphalt is in the homogeneously dispersed phase. The combination of these three constituent materials yields epoxy asphalt in which SBS, epoxy resin and asphalt are combined to result in a material that has properties different from those of the three constituents. The morphologies of epoxy asphalt indicate that solubilizer can improve remarkably the compatibility between epoxy resin and asphalt. Furthermore, SBS promotes the polymer's continuous phase, forming in epoxy asphalt. However, the viscosity of modified asphalt increases with the increase of SBS contents, and the SBS is made of thermoplastic materials. The modified asphalt softens or flows when the temperature is high enough. It is disadvantage for epoxy asphalt application. Thus, the contents of SBS should be controlled in epoxy asphalt. The experimental results indicate that the contents of SBS cannot more than 5 wt%.

Mechanical Properties

Fig. 4 shows the effect of solubilizer content on the mechanical properties of epoxy asphalt with 30 wt% epoxy resin. The results indicate that the tensile strength of epoxy asphalt increases when the solubilizer content increases, and the elongation at break decreases while the solubilizer content increases. The tensile strength of epoxy asphalt without solubilizer is only 0.91 MPa, but it is 1.25 MPa when 2.0 wt% solubilizer was added. The elongation at break of epoxy asphalt without solubilizer is 60%, and it is 40% when 2.5 wt% solubilizer was added. This may be attributed to the homogenous mixture of epoxy resin and asphalt when solubilizer is added.

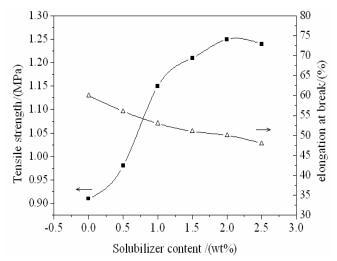


Fig. 4. Effect of Solubilizer Content on Mechanical Properties of Epoxy Asphalt with 30wt% Epoxy Resin.

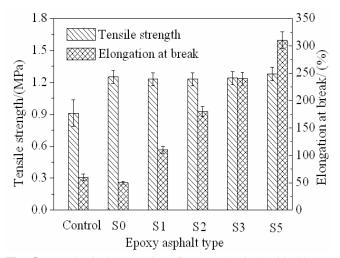


Fig. 5. Mechanical Properties of Epoxy Asphalt with 30 wt% Epoxy Resin Containing Solubilizer and Different Contents of SBS (S0, S1, S2, S3 and S5 Denotes Epoxy Asphalt Containing Solubilizer, 1 wt% SBS + Solubilizer, 2 wt% SBS + Solubilizer, 3 wt% SBS + Solubilizer, 5 wt% SBS + Solubilizer, Respectively).

Mechanical properties of epoxy asphalt with solubilizer and different contents of SBS is shown in Fig. 5. The tensile strengths of control epoxy asphalt and the epoxy asphalt containing solubilizer are 0.91 MPa and 1.25 MPa, respectively. The results show that the tensile strength of epoxy asphalt increases when the solubilizer was added. This attributes to the homogeneous blending between epoxy resin and asphalt after solubilizer was added. SBS has small effect on the tensile strength of epoxy asphalt, but it can improve remarkably the elongation at break of epoxy asphalt. The elongation at break of control epoxy asphalt is 60%, but it is 310% when 5% SBS was blended with asphalt. The results indicate that SBS can increase and improve the elongation at break of epoxy asphalt. However, SBS can increase the viscosity of the asphalt. Considering construction process, the contents of SBS in epoxy asphalt should be limited.

Conclusions

The compatibility between epoxy resin and asphalt is a crucial problem, especially on orthotropic steel bridge deck pavement. This study successfully prepared epoxy asphalt with both good compatibility and mechanical properties. On the basis of the results of this laboratory investigation on the compatibility and mechanical properties of epoxy asphalt compared with control asphalt, the following conclusions can be drawn:

- The compatibility between epoxy resin and asphalt is bad.
 Epoxy asphalt with better compatibility and mechanical properties was prepared by incorporating solubilizer and SBS.
- (2) SBS can improve the compatibility of epoxy asphalt and alter its morphology. The interlocked continuous phase forms when SBS content increases.
- (3) SBS has small effect on the tensile strength of epoxy asphalt, but it can improve remarkably the elongation at break of epoxy asphalt.

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