Compatibilizer in Waste Tire Powder and Low-Density Polyethylene Blends and the Blends Modified Asphalt

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ABSTRACT: With the increasing ratio of waste tire powder (WTP) to low-density polyethylene (LDPE), the hardness and tensile strength of the WTP/LDPE blends decreased while the elongation at break increased. Five kinds of compatibilizers, such as maleic anhydride-grafted polyethylene (PE-*g*-MA), maleic anhydride-grafted ethylene-octene copolymer (POE-*g*-MA), maleic anhydride-grafted linear LDPE, maleic anhydride-grafted ethylene vinyl-acetate copolymer, and maleic anhydride-grafted styrene-ethylene-butylene-styrene, were incorporated to prepare WTP/LDPE blends, respectively. PE-*g*-MA and POE-*g*-MA reinforced the tensile stress and toughness of the blends. The toughness value of POE-*g*-MA incorporating blends was the highest, reached to 2032.3 MJ/m^3, while that of the control was only 1402.9 MJ/m^3. Therefore, POE-*g*-MA was selected as asphalt modifier. The toughness value reached to the highest level when the content of POE-*g*-MA was about 8%. Besides that the softening point of the modified asphalt would be higher than 60°C, whereas the content of WTP/LDPE blend was more than 5%, and the blends were mixed by stirring under the shearing speed of 3000 rpm for 20 min. Especially, when the blend content was 8.5%, the softening point arrived at 82°C, contributing to asphalt strength and elastic properties in a wide range of temperature. In addition, the swelling property of POE-*g*-MA/WTP/LDPE blend was better than that of the other compalibitizers, which indicated that POE-*g*-MA /WTP/LDPE blend was much compatible with asphalt. Also, the excellent compatibility would result in the good mechanical and processing properties of the modified asphalt. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 123: 485–492, 2012

Key words: waste tire powder; low-density polyethylene; compatibilization; mechanical properties

INTRODUCTION

With the rapid development of the automobile industry, the number of autos increased sharply, and so World is facing the environmental problem related to the disposal of large-scale waste tires.[1] So, waste tire as massive waste is more and more attractive throughout the world.[2–5] Nowadays, comprehensive utilizations of waste tire are designed as state policy.[6–8] Among them, the road pavement is the mainly application of waste tire powder (WTP).[9] That is because this road pavement material WTP will enhance the durability and functional properties of the road, no matter in dry or wet mixing method.[1] Here, we must mention that "durability" of WTP-paved road means better wear and ultraviolet resistance than that of butylene-styrene copolymer.[7–11] Furthermore, WTP-paved road contributes the road with perfect noise reduction.[1,11] Accordingly, WTP has been recognized as a superior resource instead of solid massive waste. However, there are still some problems in using WTP for road pavement.[1,12] For example, in the dry-mixing processing, WTP is difficult to mix uniformity with asphalt and stone, so it will cost much time and energy.[1] On the other hand, in the wet-mixing method, the low compatibility between WTP and asphalt leads to poor storage stability at high temperature, which results in segregation of the mixture.[13] Third, the content of WTP is also an important factor, which influences the operation. For instance, when the content of WTP in asphalt is lower than 20%, the properties of modified asphalt at high in-service temperature are not satisfactory; while the content is higher than 20%, the viscosity will be too large to be paved in road.[12]

Polyethylene (PE) has been found to be one of the most effective polymer additives for the modification of paving asphalt, because it minimizes low temperature cracking and reduces rutting at elevated

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seasonal temperatures under heavy loads.[13] Accordingly, low-density PE (LDPE) has been successfully applied for road pavement at high temperature in south China. So, LDPE is blended with WTP, which would attribute asphalt with good service at elevated seasonal or low temperature. In the article, the properties of WTP/LDPE blends will be studied. Meanwhile, five compatibilizers are selected to improve the compatibility between WTP and LDPE separately, which are maleic anhydride-grafted PE (PE-*g*-MA), maleic anhydride-grafted ethylene-octene copolymer (POE-*g*-MA), maleic anhydride-grafted linear LDPE (LLDPE-*g*-MA), maleic anhydride-grafted ethylene vinyl-acetate copolymer (EVA-*g*-MA), and maleic anhydride-grafted styrene-ethylene-butylene-styrene (SEBS-*g*-MA). The effects of compatibilizers on the properties of WTP/LDPE blends will then be studied. The group with the best properties will be optimized as the modifier of asphalt and studied in further.

MATERIALS AND EXPERIMENTAL

Materials

Materials used in experiment are listed in Table I.

TABLE I
Materials Used in Experiment

Materials	Grade	Origin
WTP	WTRPα (60 mesh)	Shanghai Xiaoyou Rubber
LDPE	N220	Shanghai Jinshan Petroleum & Chemical
LLDPE-*g*-MA	220AA	Shanghai Shaike Petroleum & Chemical
PE-*g*-MA	–	Shanghai Shaike Petroleum & Chemical
EVA-*g*-MA	–	Beijing Organic Agents
POE-*g*-MA	–	Beijing Organic Agents
SEBS-*g*-MA	1901	Beijing Organic Agents
Asphalt	90#	Zhenhai Petroleum Asphalt Factory

Experimental

Preparation of WTP/LDPE blends

First, WTP was mixed with LDPE and compatibilizer in HAAKE rheometer for 10 min at temperature of 120°C, speed of 60 r/min. Then the mixture was transferred to a two-mill for further mixing at temperature of 120°C for 3 min.

Preparation of modified asphalt

All the modified asphalts were prepared using a high shear mixer (made by Fluko Machine, China) at 170°C and a shearing speed of 3000 rpm, and the shearing time was 20 min. First, 200 g asphalt was heated to become a fluid in an iron container, then on reaching about 170°C, WTP, LDPE or WTP/LDPE compound was added to the asphalts

Mechanical properties test

WTP/LDPE compound was first molded for 120 s at 165°C under pressure of 0.5 MPa until the mixture is hot to reach a fluid state, continuously molded for 300 s under pressure of 15 MPa, and then transferred to room temperature and molded for 200 min under a pressure of 10 MPa. Dumbbell specimens were cut and tested using a Galdablnisun universal testing machine at a crosshead speed of 500 mm/min according to GB/T528-1998. Hardness was measured using an indentation hardness tester according to GB/T531-1999.

High-temperature storage stability

After mixing, some of the modified asphalt was transferred into an aluminum toothpaste tube (32 mm in diameter and 160 mm in height). The tube was sealed and stored vertically in an oven at 163°C for 48 h, then cooled down to room temperature, and cut horizontally into three equal sections. The samples taken from the top and bottom sections were used to evaluate the storage stability of the WTP/LDPE modified asphalts by measuring their softening points. If the difference of the softening points between the top and the bottom sections was less than 3.5°C, and the samples was considered to have good high-temperature storage stability.

Swelling measurement

About 1 g WTP/LDPE compound was suspended in an aluminum toothpaste tube where preheated fresh asphalt had been poured. After the tube was stored in an oven at 100°C for 5 h if not mentioned especially, the sample was taken out for density and weight tests. The swelling ratio (S) was calculated according to the equation as follows:

$$S = (\rho_0 - \rho_1)/\rho_0 \qquad (1)$$

Where ρ_0 and ρ_1 are the densities of WTP/LDPE blends before and after swelling, respectively.

Density test

Swelled blend was taken out from the asphalt for density test (ASTM C1166, test method for vulcanized rubber after getting rid of the asphalt adhesive to the blends).

TABLE II
Effect of Compatibilizer on Properties of WTP/LDPE Blends

Properties	*Control	LLDPE	PE	EVA	POE	SEBS
Tensile strength (MPa)	8.09	8.09	8.86	7.65	8.41	7.79
Elongation at break (%)	233.8	249	287	320	339	279
Modulus at 100% (MPa)	5.93	6.06	5.89	4.72	4.93	4.99
Modulus at 300% (MPa)	–	–	–	7.52	8.06	–
Deformation set (%)	41	46	52	60	66	39
Shore A hardness	90	91	90	89	89	89
Toughness (MJ/m^3)	1402.9	1521.0	1840.9	1728.4	2032.3	1532.3

Toughness

Toughness was measured from the area under the stress–strain curve.[5]

Morphological analysis

A small drop of the asphalt was placed between two heated microscopic glass slides and squashed to form a thin film. The morphology of the asphalt was observed under an optical microscope (Leica, Germany) with a magnification of 400 times.

RESULTS AND DISCUSSION

Effect of compatibilizers on the properties of WTP/LDPE blends

WTP is vulcanized rubber powder, which shows poor compatibility with PE.[10] So, it is necessary to improve the compatibility between WTP and LDPE. The effect of five compatibilizers on the properties of WTP/LDPE blends is shown in Table II. According to Table II, the tensile strength of the PE-*g*-MA/WTP/LDPE and POE-*g*-MA/WTP/LDPE blends increases to some extent, while the tensile strength of EVA-*g*-MA/WTP/LDPE and SEBS-*g*-MA/WTP/LDPE decrease, compared with that of the control. The tensile strength of the LLDPE blend is unchanged from that of the control, indicating that LLDPE-*g*-MA has little influence on the tensile strength the WTP/LDPE blend, which is interesting phenomenon needed for further study. The elongation (at break) of the WTP/LDPE blends increases, incorporating anyone of the five compatibilizers into the blends. Especially, the one incorporated with POE-*g*-MA, its elongation increase from 233.8% (the control) to 339%. Deformation set of all the blends mixed with the compatibilizers increases except that of the SEBS-*g*-MA blend, and the deformation set of the POE-*g*-MA blend increases by 25, when compared with that of the control. The compatibilizers have little influence on Shore-A hardness of the blends. The toughness of the blends increases since introducing the compatibilizers, and the toughness of the WTP/LDPE blend with compatibilizers increases in the following sequence: LLDPE-*g*-MA, SEBS-*g*-MA, EVA-*g*-MA, PE-*g*-MA, and POE-*g*-MA. The toughness of the POE-*g*-MA is increased by 44.9%, from 1402.9 to 2032.3 MJ/m^3 of the control.

According to the above analysis, POE-*g*-MA has contributed to the greatest improvement of the properties of the WTP/LDPE blends including tensile strength, elongation at break, deformation set, and toughness, indicating that incorporation of POE-*g*-MA might lead to the best compatibility between WTP and LDPE. So, POE-*g*-MA should be selected as the agent to incorporate with WTP and LDPE for further asphalt modification.

Effect of POE-*g*-MA on the properties of WTP/LDPE blends

Compatibilizers perhaps have some influence on the properties of blends, so it is necessary to study the effect of POE-*g*-MA on the mechanical properties of the WTP/LDPE blends. The effect of POE-*g*-MA on the properties of WTP/LDPE blends is shown in Table III. POE-*g*-MA exhibited higher tensile strength

TABLE III
Effect of POE-*g*-MA on the Properties of WTP/LDPE Blends

	POE-*g*-MA content					
Properties	0	2	4	6	8	10
Tensile strength (MPa)	8.09	8.86	8.43	9.00	9.06	9.31
Elongation at break (%)	233.8	225.4	222.5	219.8	240.5	231.7
Modulus at 100% (MPa)	5.93	6.50	5.80	6.28	6.24	6.20
Deformation set (%)	41	55	45	52	51	61
Shore A hardness	90	91	90	89	91	88
Toughness (MJ/m^3)	1402.9	1465.2	1300.5	1392.5	1560.6	1411.4

TABLE IV
Effect of WTP/LDPE Ratio on the Properties of POE-*g*-MA/WTP/LDPE Blends

Properties	WTP/LDPE ratio				
	50 : 50	60 : 40	70 : 30	80 : 20	90 : 10
Tensile strength (MPa)	9.40	8.85	9.26	8.47	7.31
Elongation at break (%)	209.6	214.2	220.8	195.7	178.0
Modulus at 100% (MPa)	7.89	6.86	6.38	5.62	4.95
Deformation set (%)	98	77	71	38	25
Shore A hardness	92	90	88	85	81
Toughness (MJ/m^3)	1615.2	1435.0	1420.2	1062.0	797.6

and deformation set, lower elongation at break except at 8 phr, highest toughness at 8 phr, when compared with the control. As a result, POE-*g*-MA/WTP/LDPE (8/70/30) blend should be a modifier for asphalt.

Effect of WTP/LDPE ratio on the properties of POE-*g*-MA/WTP/LDPE blends

Different ratios of WTP to LDPE may have influence on the properties of POE-*g*-MA/WTP/LDPE blends. The effect of rubber/plastic ratio on the properties of POE-*g*-MA/WTP/LDPE was studied, as shown in Table IV. The results show that the tensile strength decreased when compared with WTP content of 50, modulus at 100%, deformation set, shore A toughness, and toughness decreased with increasing WTP content. Otherwise, elongation at break increased in the range of WTP content from 50 to 70, decreased from 70 to 90, indicating that the mechanical property is the highest when the ratio of WTP to LDPE is 70/30. Taking account of toughness, the property was the best when the ratio of WTP to LDPE was 50/50. However, LDPE is much more expensive than WTP. Combined with cost considerations, 70/30, the ratio of WTP to LDPE, should be chosen for further study for asphalt modification.

Swelling behavior of WTP/LDPE blends in asphalt

The swelling behavior of the rubber/plastic blends in asphalt has been adopted to investigate the compatibility between the blends and asphalt.[14] Five compatibilizers are respectively incorporated into WTP/LDPE to obtain blends, and the blends are further immersed in asphalt to evaluate the swelling behavior of the blends in asphalt, as shown in Figure 1. The blends can absorb the light component of the asphalt in the process of swelling, and the swelling blends exhibit lower density when compared with the nonswelling one. Lower density means better swelling properties. In Figure 2, *S* is swelling ratio, and the bigger value of the *S* indicates that the blend is more easily swelled in the asphalt. Five kinds of compatibilizers are, respectively, mixed with WTP and LDPE

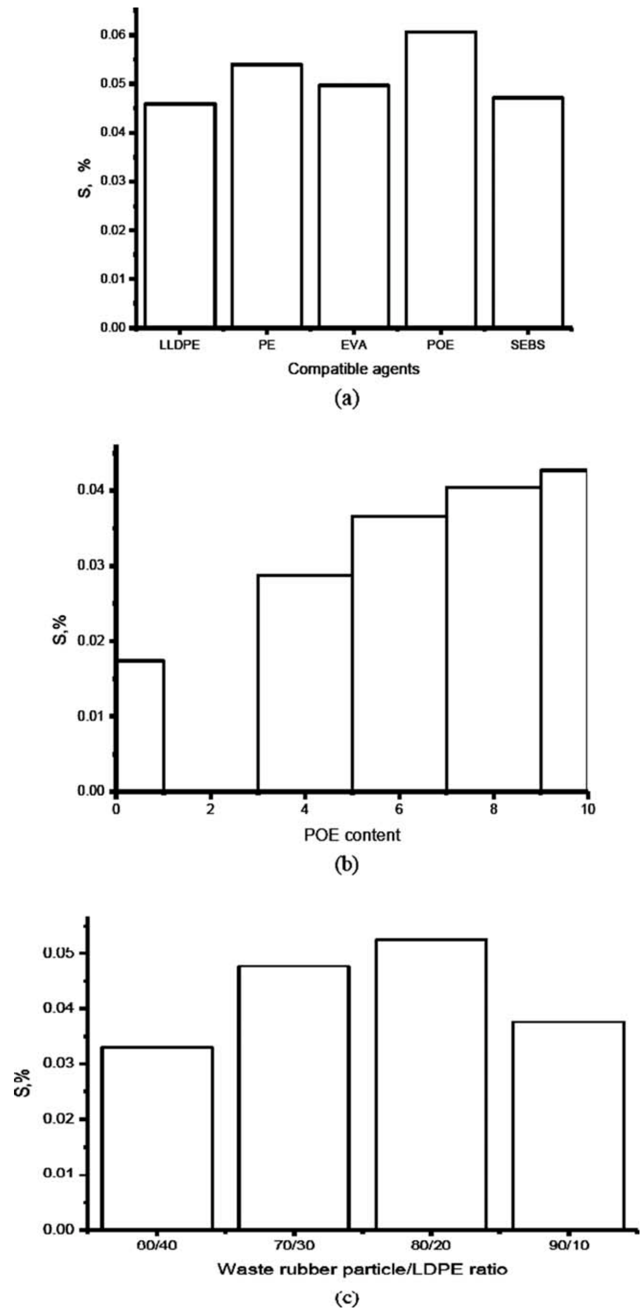

Figure 1 Effect of compatibilizer on the swelling behavior of WTP/LDPE blend.

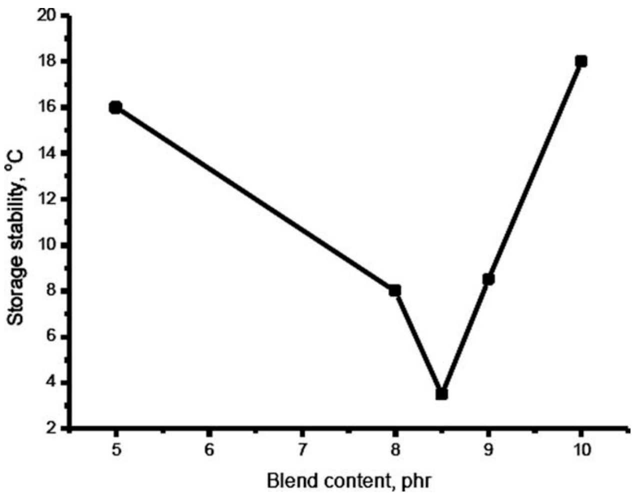

Figure 2 High-temperature storage stability of PMA.

to obscure the blends and the blends swelled in the asphalt, as shown in Figure 1(a). The results show that the swelling level of the compatibilizers to WTP/LDPE blends increased as following sequence: LLDPE-*g*-MA, SEBS-*g*-MA, EVA-*g*-MA, PE-*g*-MA, and POE-*g*-MA, which is consistent with the sequence according to the toughness. As a result, the more compatible between WTP and LDPE, the higher toughness of the blend. The effect of POE-*g*-MA content on the swelling property of WTP/LDPE blend in the asphalt is shown in Figure 1(b). The result shows that the swelling ratio of the blends increased with increasing POE-*g*-MA content, indicating that POE-*g*-MA has enhanced the compatibility between WTP/LDPE blend and asphalt, and the compatibility improved with increasing POE-*g*-MA content. The effect of WTP/LDPE ratio on the swelling property of POE-*g*-MA/WTP/LDPE blend in the asphalt is shown in Figure 1(c). The result shows that the swelling ratios of the blends with WTP to LDPE ratio of 70/30 and 80/20 are higher than those of 60/40 and 90/10, indicating that the ratio of WTP to LDPE should be in a proper range, where POE-*g*-MA/WTP/LDPE blend is rather more compatible with asphalt.

WTP/LDPE blends modified asphalt

As mentioned above, the mechanical properties of the POE-*g*-MA/WTP/LDPE (8/70/30) blend is rather good. So, the blend is further mixed with asphalt to prepare polymer-modified asphalt (PMA). The properties of PMA are shown in Table V. With increasing the blend content, PMA presents higher softening point, viscosity, and lower needle penetration, ductility. The softening point increases from 61.5 to 82°C, and the viscosity increases from 2040 to 2647 cp as the blend content increases from 8 to 8.5, indicating that the blends have significantly improved the high in-service temperature of the asphalt. High-temperature storage stability is shown in Figure 2. The results show that the PMA presents good high-temperature storage stability at the blend content of 8.5, otherwise, the PMA are unstable at high temperature. The effect of the blend on the morphology of the PMA is shown in Figure 3. The polymer particles are irregularly dispersed in asphalt matrix when the blend content is less than 8. With increasing the blend content to 8.5, the blend particles link together and scatter in the asphalt, and the modified asphalt presents bicontinuous phase, as shown in Figure 3(c). With the further addition of the polymer content, the polymer accumulates and forms a continuous phase.

As a result, WTP/LDPE blend is very useful for road pavement to improve the main distress associate with asphalt: viscous properties at high in-service temperature that favor permanent deformation. Especially, the softening point increased dramatically when the blend content increased from 8 to 8.5. The phenomenon might be explained that the blend absorbs some part of light component of the asphalt and the absorbing blend intends to form a continuous phase at a critical content.

Mechanism of storage stability

The high-temperature storage stability of WTP/LDPE blends modified asphalt with different blends content under medium mixing condition are tested, as shown in Figure 2. So, two problems occurred.

TABLE V
Properties of PMA

Properties	Blend content (phr)				
	5	8	8.5	9	10
Softening point (°C)	60.5	61.5	82	88	91.5
Needle penetration (dmm)	66.9	38.1	29.1	35.5	32.4
Ductility (mm)	8.6	6.7	4.5	3.5	4.1
Viscosity (cp)	1439	2040	2647	3395	6121
Upper softening point (°C)	67	68	84.5	89.5	93
Bottom softening point (°C)	51	60	81	81	75

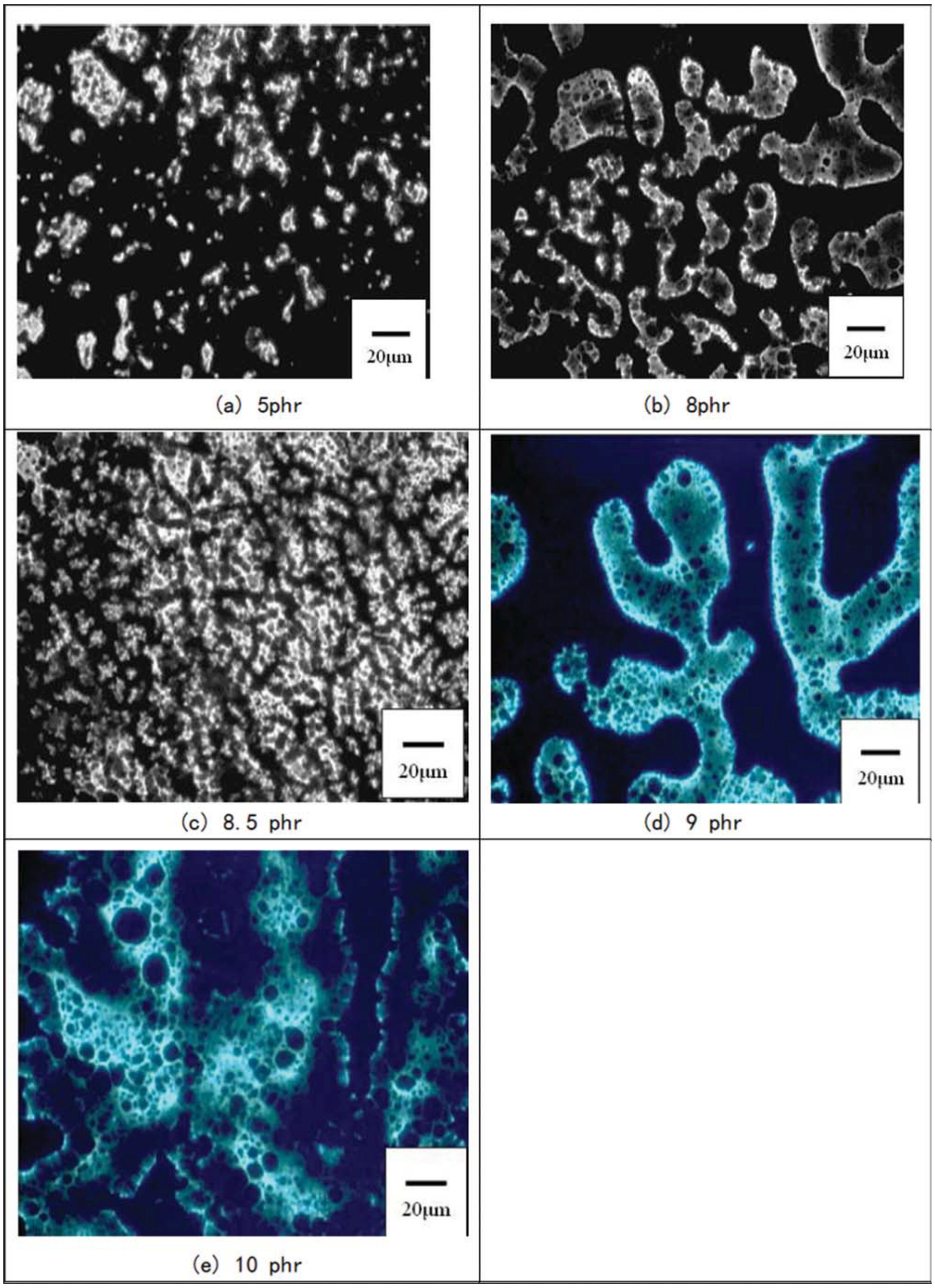

Figure 3 Morphology of PMA. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

One is storage stability and the other is a proper blends content. There might be two kinds of explanations as follows. First, asphalt is traditionally considered as a dynamic colloid system consisting of a suspension of high-molecular-weight asphaltene micelles dispersed in a lower molecular weight oily medium (maltenes).[15] The PMAs may have a tendency to separate into two phases, one is a polymer-rich phase and the other one asphalt-rich phase, because the introduction of any polymer will disturb the dynamic equilibrium and reduce the homogeneity of the asphalt system.[16–20] The case is utmost serious under quiescent conditions at high temperature.[21]

WTP/LDPE-modified asphalt can be considered as a suspended system. For the suspended system, the particles in the liquid with the buoyancy force and gravitational force, and the falling velocity of the particles in the system follow Stoke's law,[21]

$$V = 2(\rho_0 - \rho_1)gr^2/9\eta \qquad (2)$$

where ρ_0 is the density of asphalt, ρ_1 is the density of WTP/LDPE blend, g is the gravitational force

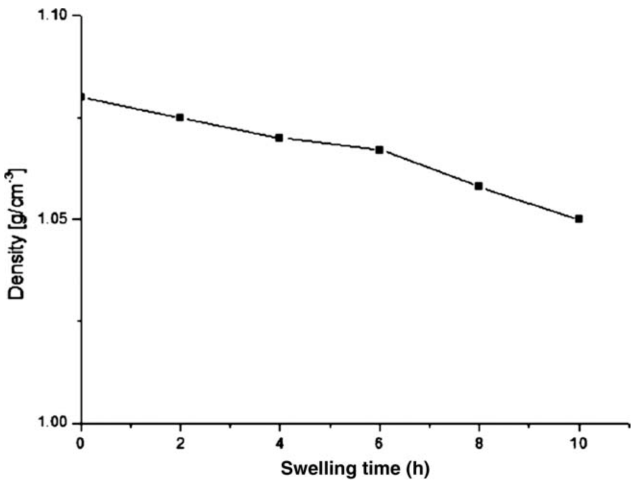

Figure 4 Density of WTP/LDPE/POE-*g*-MA compound swelling in asphalt varied with time.

constant, r is the average radius of the blend particles, and η is the viscosity of the modified asphalt.

To prevent the phase separation of the blend from asphalt, a critical way is to reduce the falling velocity of the particles. As shown in eq. (1), there are two methods to reduce the falling velocity, one is to reduce the particle size and the other is to decrease the density difference.

WTP is a particulate vulcanizate consisting mainly of rubber hydrocarbon and fillers such as carbon black with active functional groups on its surface. POE-*g*-MA is a compatibilizer with functional groups. Chemical or physical interactions with LDPE can form during processing when WTP and POE-*g*-MA incorporated with LDPE.

The density of the LDPE is 0.91 g/cm^3, and the density of asphalt here is 1.02 g/cm^3 at room temperature. The density of WTP is around 1.2 g/cm^3 at room temperature. When the WTP attached to LDPE, the density difference is decreased and the force for driving separation becomes zero at a certain content of WTP, so the high-temperature storage stability is improved. The swelling properties of the WTP/LDPE/POE-*g*-MA compound in the asphalt is shown in Figure 4. The density of the compound was 1.06 g/cm^3 at the beginning stage of swelling. With the swelling proceeding, the density of the compound was decreased slowly. According to the trend of the curve, the density of the compound had a tendency to reach the density of the asphalt, which implied that the density difference between the compound and asphalt is minimized. So the system reached in a stable condition.

There might be another explanation. When LDPE is compounded with WTP and POE-*g*-MA, WTP particles can attach to the POE-*g*-MA, and the POE-*g*-MA can attach to the LDPE, and change the polarity of the WTP/LDPE/POE-*g*-MA compound, as shown in Figure 5(a). Meanwhile, the compound can absorb the light component of the asphalt, and POE-*g*-MA acts as interfacial agent related between the blend and the asphalt including asphaltene, as shown in Figure 5(b). POE-*g*-MA with different extents of polarity results in good dispersion of polymer additive into the asphalt phase and affects the certain degree of the phase separation of asphalt blends.[12] The storage-stability of the compound-modified asphalt would be obtained by equalizing polarity differences between asphalt and LDPE by WTP and POE-*g*-MA.

CONCLUSIONS

The hardness and tensile strength of the WTP/LDPE blends decrease and elongation at break increases with increasing the ratio of WTP to LDPE.

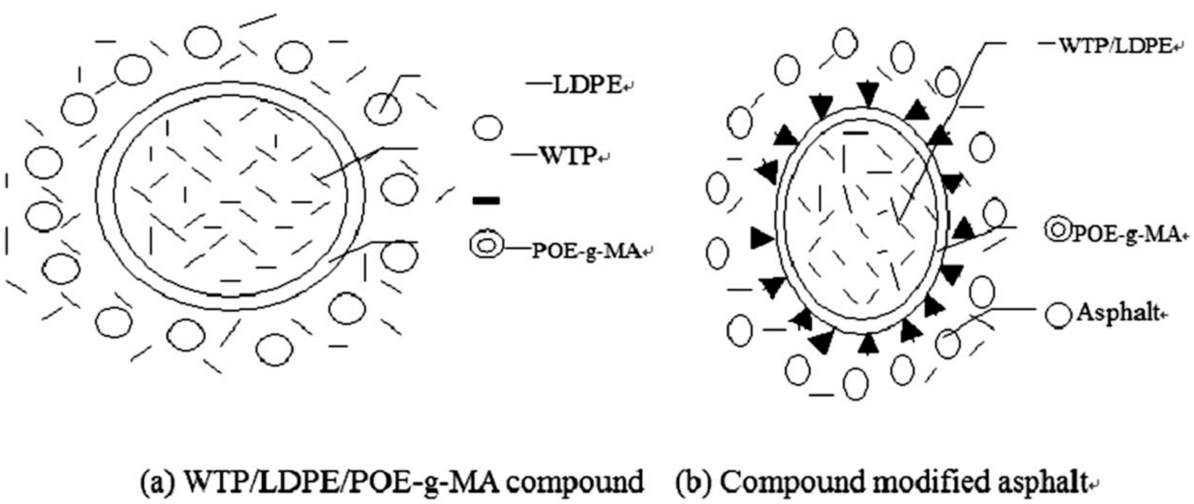

Figure 5 Scheme of WTP/LDPE/POE-*g*-MA blending with asphalt.

Improving the compatibility between WTP and LDPE is a good method to reinforce the blend properties. Five compatibilizers, such as PE-*g*-MA, POE-*g*-MA, LLDPE-*g*-MA, EVA-*g*-MA, and SEBS-*g*-MA, are compounded to prepare WTP/LDPE blends, respectively. PE-*g*-MA and POE-*g*-MA reinforce the tensile stress and toughness of the blends to some extent. Among the five compatibilizers, the toughness value of the POE-*g*-MA blend is the biggest, reaches to 2032.3 MJ/m^3, while that of the control is 1402.9 MJ/m^3. POE-*g*-MA is selected as asphalt modifier. The toughness value of the WTP/LDPE/POE-*g*-MA blend is the biggest at POE-*g*-MA content of 8%. The softening point of the modified asphalt is more than 60°C at WTP/LDPE ratio of 70/30, at the blend content of more than 5%, POE-*g*-MA content of 8%, under shearing speed of 3000 rpm for 20 min (Traditonal condition of 5000 rpm for more than 60 min, as reported). When the blends content is 8.5%, the softening point of the blends is 82°C. So some measurements such as swelling properties should be taken to give explanation of the improvement. Swelling is to assess the compatibility between the asphalt and the WTP/LDPE blend before and after incorporating POE-*g*-MAH. Sure, whether WTP or LDPE is not compatible with asphalt. So, WTP/LDPE blend is not easily to swell in the asphalt. POE-*g*-MAH contained polar and nonpolar groups and equalized polarity difference between asphalt and WTP/LDPE. So, the WTP/LDPE blend incorporated with POE-*g*-MAH is much more easily swelled in the asphalt. The swelling property of POE-*g*-MA blend is the best among the five compalibitizers, and the blends modified asphalt with good storage stability at high temperature has been obtained. The stable storage may arise from two factors, one is that WTP decreases density difference between LDPE and asphalt and the other is that POE-*g*-MA might change the polarity of WTP/LDPE compound in asphalt, resulting in dispersion of polymer additive into the asphalt phase and affecting the certain degree of the phase separation of asphalt blends. POE-*g*-MA should be a good modifier to improve the compatibility between the asphalt and the WTP/LDPE blend.

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