Construction and Building Materials 68 (2014) 769–776

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Multiscale test research on interfacial adhesion property of cold mix asphalt

Meng Guo [a], Yiqiu Tan [a,*], Shuiwen Zhou [b]

[a] School of Transportation Science and Engineering, Harbin Institute of Technology, Harbin, Heilongjiang, China
[b] Sichuan Provincial Transport Department Highway Planning, Survey, Design And Research Institute, Chengdu, Sichuan, China

HIGHLIGHTS

- Cold-mix diluents and additives can increase surface free energy of pure asphalt.
- Limestone has the largest SFE and adhesion work to asphalt in dry condition.
- Cohesion and adhesion failure usually occurs in dry and damp condition respectively.
- A good correlation exists between macro mechanical indicators and energy indices.
- Limestone is generally better at resisting moisture damage than granite and basalt.

ARTICLE INFO

Article history:
Received 19 December 2013
Received in revised form 15 June 2014
Accepted 18 June 2014

Keywords:
Cold mix asphalt
Interfacial adhesion
Surface free energy
Multiscale test
Moisture damage

ABSTRACT

In order to establish proper evaluation methods and indicators for cold mix asphalt (CMA), various scales test methods were used to study the interfacial behavior between asphalt binder and aggregate. The surface free energy of asphalt binder was tested using sessile drop method, and the surface free energy of aggregate was measured using column wicking method. The adhesion work between asphalt and aggregate, moisture damage resistance indices were calculated using the basic surface free energy components. Macro mechanical adhesion property between asphalt binder and aggregate in two conditions (dry and damp) were obtained through the pull-off test. A freeze–thaw splitting test was conducted to verify the effectiveness of energy indices. The results show that diluents and additives used in cold mix asphalt can increase surface free energy of pure asphalt. Limestone has the largest surface free energy, and the largest adhesion work to asphalt in dry condition. Cohesion failure usually occurs in dry condition, while adhesion failure corresponds to damp condition. There are strong correlations between macro mechanical evaluation indicators and surface free energy calculated indices, which indicates that energy indices are a good method to quantize the moisture damage resistance of CMA. Limestone is generally better at resisting moisture damage than granite and basalt.

1. Introduction

Nowadays, asphalt pavement is used widely as one of main pavement styles. However, over time its performance will degenerate, and many kinds of distresses can occur, such as potholes. In southern China, many pavements are located in moist highland mountainous areas, and ice freezing usually occurs, which can easily lead to a pitted, loose surface with many potholes. In order to solve these problems, and recover the traffic capacity of road in a short time, the maintenance department usually applies cold mix asphalt (CMA) as an emergency repair. CMA has several advantages over the more common HMA, particularly the fact that it requires no heat to manufacture or lay over the pavement. As a result, such pavement has less of an environmental impact, is more cost effective, and requires less energy consumption. However, the performance of cold mix asphalt is usually worse than hot mix asphalt or warm mix asphalt because of the limited construction conditions, especially in terms of adhesion properties and moisture damage resistance. In addition, the evaluation methods and indicators related to cold mix asphalt are also not very clear and convincing.

Many researchers have done a lot of work evaluating cold mix asphalt, and they have found that there are obvious difference between CMA and HMA. Anna Abela Munyagi evaluated the proprietary cold mix asphalts available in South Africa. He found

---

* Corresponding author. Tel.: +86 15846604263.
E-mail addresses: mguo@hit.edu.cn (M. Guo), Tanyiqiu@hit.edu.cn (Y. Tan).

http://dx.doi.org/10.1016/j.conbuildmat.2014.06.031
0950-0618/

that the Indirect Tensile Strength values of all the tested cold mix asphalt were very low compared to minimum value of 800 kPa specified for Hot Mix Asphalt. And all tested cold mix asphalt were highly susceptible to rutting compared to Hot Mix Asphalt [1]. In order to solve the rutting problem of cold mix asphalt, Chavez-Valencia et al. thought that the compressive strengths of cold mix asphalt must be improved. For this reason they added polyvinyl acetate emulsion (PVAC-E) to a cationic quick set emulsified asphalt to obtain a modified asphalt emulsion that was mixed with a local aggregate in order to prepare two types of CMA. They found that the compressive strength was improved by 31% compared to the values obtained with the unmodified CMA [2]. Similarly, Benedito et al. selected fiber to reinforce CMA, and they found the addition of fiber is responsible for a small variation in mixture strength parameters, as well as for substantial drops in the mixture resilient moduli when compared to plain mixtures [3]. Al-Busaltan et al. used waste materials to mix a new CMA, and found it has superior mechanical properties compared to traditional HMA [4]. Due to the associated peculiarities with cold asphalt encompassing the presence of water, emulsion–aggregate reactivity, evolving characteristic with time and an undeveloped internal structure, CMA does not lend itself very well to investigation of the influence of material and/or process variables, such as moisture condition, on its mechanical properties [5]. Hussain et al. did research on the effects of moisture ingress on the mechanical properties of a cold-laid grave emulsion asphalt mixture by developing a vacuum moisture saturation technique. They found that the cold mixture's results were only marginally lower, even though the hot mixture had good fracture properties. The vacuum saturation treatment led to enhanced yield performance at low temperatures of both mixtures in the fracture test. The high level of moisture treatment given to the cold mixture made it behave like the hot asphalt mixture at low temperatures [6]. In terms of assessment methods for CMA, Thomas et al. presented the development of a maturity approach for the assessment of cold mix bituminous materials and its application for predicting the effects of climatic variations on in-situ mixture performance. They observed a strong correlation existing between the calculated maturity and the measured stiffness for a range of conditioning temperatures and durations thus enabling the prediction of long and short-term materials performance in situ where ambient conditions are known [7].

In order to evaluate the moisture damage resistance of asphalt mixture, most researchers focus on the experiential methods, such as immersion Marshall test and freeze–thaw splitting test. Recently, some researchers paid attention to the interface adhesion property in asphalt mixture, and they think it can explain the moisture damage mechanism substantially [8–11]. Bhasin et al. thought that physical adhesion is probably the adhesion component (as opposed to the chemical interactions and mechanical interlocking) that predominantly contributes to the overall adhesion of the asphalt–aggregate systems. They found that surface free energy is an effective method to study the adhesion property, and they successfully obtained the surface free energy parameters for asphalt binder and aggregate [9]. Following that, Tan and Guo used different methods to test surface free energy parameters of asphalt binder and fillers, and calculated the adhesion work of asphalt mastic [12]. Based on surface free energy, Bhasin et al. proposed two parameters to evaluate the moisture damage resistance of asphalt mixture [9,13]. In the study of asphalt mixture, there are two main methods of evaluating moisture damage resistance. One is the traditional experimental method, and the other is quantized surface free energy method. However, the relationship between the two scales is not very clear currently.

Our research focused on the adhesion property between asphalt and aggregate based on surface free energy theory and mechanical pull-off test, and analyzed the relationship among different scales adhesion properties. This is significant for the establishment of evaluation methods and indicators for cold mix asphalt.

2. Materials and test methods

2.1. Experimental materials

2.1.1. Asphalt binder

The grade of asphalt binder used in our research is 90. Its basic properties are shown in Table 1.

2.1.2. Aggregate and filler

The chemical constituent and surface morphology both play an important role in enhancing the adhesion property between asphalt binder and aggregate. Generally, alkaline aggregate has better adhesion property than acidic aggregate. In our research, we selected three kinds of aggregates: granite, limestone and basalts. Fillers were made from the above aggregates through magnetic milling.

2.1.3. Diluents

The viscosity of asphalt binder at low temperature is very high, so some diluents must be used to decrease its viscosity to improve the workability. The diluents used in our research include 0# diesel, 90# gasoline, ordinary kerosene and aviation kerosene. Their basic properties are shown in Table 2.

2.1.4. Additives

The diluents can make the asphalt binder much softer, but at the same time they can also change other properties of asphalt binder so that the asphalt mixture cannot meet the actual requirements. So we added some additives to improve the performance of cold mix asphalt. Generally, the additives can improve the adhesion property between asphalt binder and aggregate to enhance the moisture damage resistance ability. It can also increase the early strength of CMA while improving the workability. We selected four typical kinds of additives, which are KN, LB, GS and SJ.

2.1.5. Grading of mixture

The grading of mixture used in our research is shown in Fig. 1, and its optimum asphalt content is 5.3%.

2.2. Laboratory testing methods

2.2.1. Sessile drop method

Sessile drop method used in our research is similar with literature [12], and the surface free energy parameters of three kinds of test liquids are shown in Table 3.

In order to study the effect of diluent types, additive types, diluent content, and additive content on surface free energy of asphalt binder, we applied orthogonal experiment design method. The design plan is shown in Table 4. The test results are shown in Table 5.

Table 1
The basic properties of 90# asphalt binder.

	Items	Units	Results	Requirements
Before aging	Penetration (25 °C, 100 g, 5s)	0.1 mm	83.3	80∼100
	Penetration Index	–	0.47	−1.5∼ + 1.0
	Ductility	cm	133	⩾100
	Soft point	°C	51.4	44
	Density (15 °C)	g/cm^3	1.03	–
	Flash point (COC)	°C	310	⩾245
After aging	Mass loss	%	0.75	⩽±0.8
	Residual ductility, (5 cm/min, 15 °C)	cm	22	⩾20
	Residual penetration ratio (25 °C, 100 g, 5s)	%	66.8	⩾57

Table 2
The basic properties of diluents.

Types	Dynamic viscosity (20 °C, Pa s)	Flash point (°C)	Fire point (°C)	Evaporation speed
0# diesel	2.55–6.8	⩾55	220	Moderation
90# gasoline	–	−50	427	Extremely fast
Ordinary kerosene	–	45	80–84	Fast
Aviation kerosene	–	38	–	Very fast

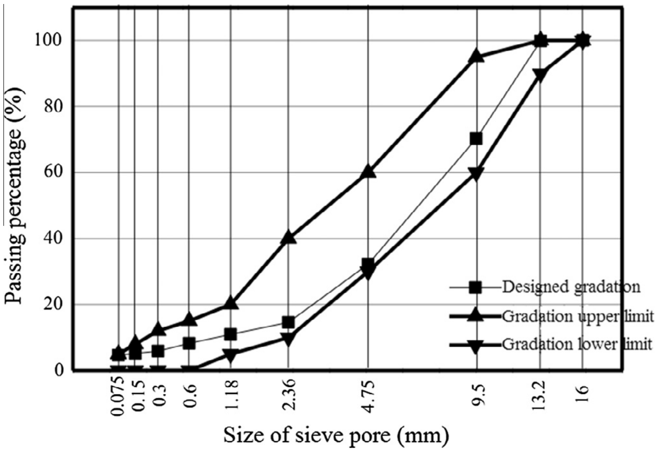

Fig. 1. The mixture grading.

2.2.2. Column wicking method

Wei and Zhang used the sessile drop method to characterize surface free energy of aggregate [14], however, this methods need the aggregate surface to be treated to be smooth, which can change the surface property at natural condition. So the SFE parameters of aggregate obtained using this method may not be the actual value in asphalt mixture. In order to overcome this disadvantage, our research chose column wicking method to characterize the SFE parameters of aggregate. The SFE is the inherent attribute of aggregate, which will not depend on geometrical shape, so the value from powder can reflect the actual state of aggregate in asphalt mixture. The detailed test procedure can be seen in literature [12].

2.2.3. Pull-off test

Up to now, there have been mainly three kinds of tests to study the interfacial adhesion property of asphalt mixture. They are the dynamic mechanical analyzer (DMA) tension test, the pull-off test, and the asphalt–aggregate shear test. Considering the importance of interaction between surface texture, contour, angularity of aggregate, and asphalt bind, we selected pull-off test to test the adhesion property between asphalt mastic and aggregate. The test procedure is as follows:

(1) We selected several aggregates with similar shape and similar surface texture, and then drew two lines standing for the deep immersing in asphalt mastic and glue, respectively (Fig. 2-a).
(2) We filled some glue in the mold, and then put the aggregate into glue to the lower line and waited until the glue solidified completely (Fig. 2-b).
(3) We smeared the vaseline on the inwall of sleeve, and assembled the sleeve and the prepared aggregate to make the sleeve support the aggregate (Fig. 2-c).
(4) We put a certain amount of heated asphalt mastic into the other mold (Fig. 2-d).
(5) We assembled the two molds together, and adjusted the bolt to make the aggregate immerse in the asphalt mastic to the other deep line (Fig. 2-e).
(6) After the sample cooled down, we put it in the temperature-control oven at test temperature for 4 hours. Then we took out the sample, unloaded the supporting cylinder, and fixed the bolt of sample on the MTS lower chuck (Fig. 2-f). Following that, we unloaded the cylinder fixing aggregate mold (Fig. 2-g). Finally, we used the switched bolt to fix the aggregate to the upper chuck of MTS device (Fig. 2-h).

Table 4
Orthogonal experiment design for cold mix asphalt binder.

Serial number	Diluent types	Additive types	Diluent content (%)	Additive content (%)
1	0# diesel	KN	10	1
2	90# gasoline	LB	15	2
3	Ordinary kerosene	GS	20	3
4	Aviation kerosene	SJ	25	4
5	0# diesel	KN	25	2
6	90# gasoline	LB	20	1
7	Ordinary kerosene	GS	15	4
8	Aviation kerosene	SJ	10	3
9	0# diesel	KN	15	3
10	90# gasoline	LB	10	4
11	Ordinary kerosene	GS	25	1
12	Aviation kerosene	SJ	20	2
13	0# diesel	KN	20	4
14	90# gasoline	LB	25	3
15	Ordinary kerosene	GS	10	2
16	Aviation kerosene	SJ	15	1

(7) We ran the tension test at a 50 mm/min speed (Fig. 2-i). After the tests finished, we measured the bare area, and analyzed the damage style.

In order to verify the effectiveness of parameter ER_1 and ER_2 on evaluating moisture damage resistance of CMA, this section selected six combinations to conduct the pull-off test (Table 6).

2.2.4. Freeze–thaw splitting test

In order to simulate the weather condition of southern China, the freeze–thaw procedure was set as follows: (1) Put the sample into vacuum water for 15 min, and then transfer it to water under normal pressure for 30 min. (2) Keep the sample in −5 °C atmosphere for 12 h. (3) Transfer the sample in 5 °C water for 12 h. (4) Make each sample go through the above steps three times, and then test its performance.

In this section, three combinations were selected, which were 14# asphalt + granite, 10# asphalt + limestone, and 15# asphalt + granite. The compaction method is Mashall compaction. Each sample had a 63.5 ± 1.3 mm height.

3. Results and discussion

3.1. The surface free energy of asphalt binder

It can be seen from Table 5 that the total surface free energy of all the asphalt added diluents and additives increase compared with original pure asphalt, although some components decrease.

Table 3
The SFE parameters of test liquids.

Test liquids	γ_L (mJ/m^2)	γ_L^{LW} (mJ/m^2)	γ_L^{AB} (mJ/m^2)	γ_L^{+} (mJ/m^2)	γ_L^{-} (mJ/m^2)
Distilled water	72.8	21.8	51	25.5	25.5
Glycerinum	64	34	30	3.92	57.4
Glycol	48	29	19	1.92	47

Table 5
Test result of surface free energy of asphalt binder for various combinations.

Serial number	γ_L (mJ/m^2)	γ_L^{LW} (mJ/m^2)	γ_L^{AB} (mJ/m^2)	γ_L^- (mJ/m^2)	γ_L^+ (mJ/m^2)
Pure asphalt	14.99	13.99	1.60	0.15	4.38
1	17.49	15.77	1.72	0.19	3.98
2	20.99	20.74	0.25	0.01	3.04
3	18.28	17.11	1.17	0.07	5.21
4	23.77	23.14	0.63	0.06	1.74
5	29.04	26.56	2.48	0.42	3.65
6	33.31	31.95	1.36	0.18	2.61
7	25.16	25.09	0.07	0.00	1.39
8	23.07	22.12	0.94	0.05	4.35
9	20.07	19.66	0.41	0.01	4.12
10	23.35	22.64	0.71	0.03	4.88
11	21.19	20.96	0.24	0.00	7.04
12	18.52	18.10	0.43	0.01	7.31
13	21.77	20.54	1.23	0.16	2.33
14	18.10	16.86	1.24	0.06	6.93
15	23.51	23.48	0.03	0.00	0.24
16	24.84	24.66	0.18	0.01	1.21

In order to study the sensitivity of different factors on SFE of asphalt binder, variance analysis were done (Table 7).

In Table 7, *P*-value can reflect significance level, the smaller *P*-values represent the more significant effects. It can be seen from Table 7 that the significant rank of the factors effecting SFE is diluent types > additives content > diluent content > additives types.

3.2. Surface free energy of aggregate

In this section, hexane was used to steep granite, basalt, and limestone, and an X^2–t relationship was obtained. Using slope and parameters related to hexane, the effective radii of capillary were calculated as follows: $R_{\text{Granite}} = 3.3 \pm 0.30$um, $R_{\text{Basalt}} = 3.3 \pm 0.30$um, $R_{\text{Limestone}} = 3.3 \pm 0.30$um, Following this step, diiodomethane, toluene, chloroform were used to steep granite, basalt, and limestone. The calculated surface free energy and its components are shown in Table 8.

Table 6
Six combinations undergoing the pull-off test.

Combinations	Adhesion work (mJ/m^2)		ER$_1$	ER$_2$
	W_{as}	W_{asw}^{wet}		
1# + Limestone	48.34	49.22	0.98	0.27
14# + Granite	44.46	58.49	0.76	0.14
10# + Limestone	56.00	50.11	1.12	0.19
9# + Basalt	47.21	65.89	0.72	0.11
6# + Limestone	62.15	51.29	1.21	0.09
15# + Granite	46.28	77.15	0.60	0.01

Table 7
Variance analysis of factors affecting SFE of asphalt binder.

Indicator	Factors	SS	Df	MS	P
Surface free energy	Diluent types	140.256	3	46.752	0.007
	Additive types	9.424	3	3.141	0.222
	Diluent content	43.916	3	14.639	0.034
	Additive content	66.605	3	22.203	0.019
	e	3.548	3	1.183	0.007
	Total	263.749	15	–	–

It can be seen from Table 8 that surface free energy of the aggregates are very different, while Van der Waals' forces have small difference, so the difference is mainly results from Levis acid and alkali force. The surface free energy and its components of limestone are larger than the other two, which agrees with Yiqiu Tan and Meng Guo's conclusion [12]. Granite aggregate has the lowest surface free energy and components.

3.3. Adhesion work between asphalt and aggregate

According to literature [12], the adhesion work between asphalt and aggregate can be calculated using the following equations.

Dry condition:

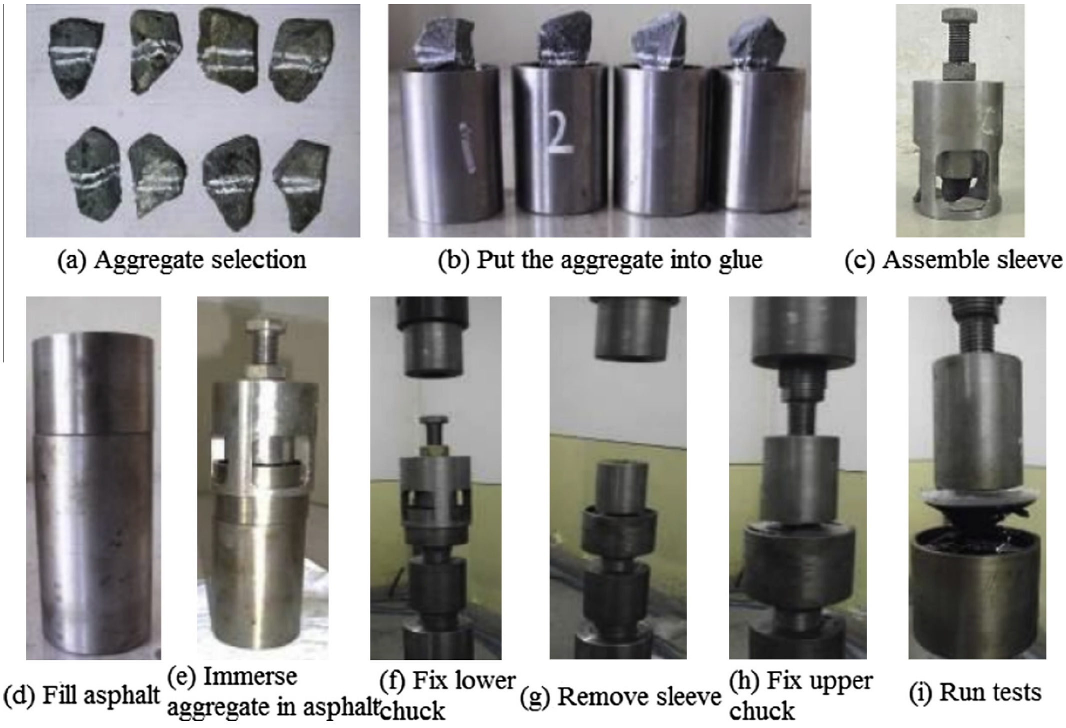
(a) Aggregate selection
(b) Put the aggregate into glue
(c) Assemble sleeve
(d) Fill asphalt
(e) Immerse aggregate in asphalt
(f) Fix lower chuck
(g) Remove sleeve
(h) Fix upper chuck
(i) Run tests

Fig. 2. The procedure of pull-off test.

Table 8
Surface free energy of aggregate and its components.

Aggregate	γ_s (mJ/m^2)	γ_s^{LW} (mJ/m^2)	γ_s^{AB} (mJ/m^2)	γ_s^{+} (mJ/m^2)	γ_s^{-} (mJ/m^2)
Granite	19.10	18.20	0.90	0.05	4.29
Basalt	23.81	21.71	2.10	0.93	1.19
Limestone	28.94	21.91	7.03	1.99	6.21

Table 9
Adhesion work results.

Asphalt	Granite		Basalt		Limestone	
	W_{as}	W_{asw}^{wet}	W_{as}	W_{asw}^{wet}	W_{as}	W_{asw}^{wet}
1	39.40	61.58	40.83	63.28	45.76	49.08
2	42.20	61.93	43.18	63.19	48.34	49.22
3	46.38	66.84	47.06	67.80	51.52	53.13
4	44.03	61.07	45.73	63.06	50.83	49.02
5	48.17	68.85	47.62	68.58	52.28	54.10
6	53.44	60.81	53.25	60.89	59.59	48.10
7	57.01	65.00	56.29	64.56	62.15	51.29
8	49.31	71.65	48.68	71.31	52.85	56.34
9	48.82	62.88	49.86	64.20	55.06	50.27
10	45.94	64.35	47.21	65.89	51.91	51.46
11	49.47	62.44	50.81	64.05	56.00	50.11
12	48.54	59.87	51.03	62.64	56.20	48.68
13	45.69	59.20	48.42	62.21	53.52	48.17
14	46.34	65.86	46.12	65.92	51.17	51.84
15	44.46	58.49	46.90	61.21	52.22	47.40
16	46.28	77.15	44.39	75.54	47.87	59.89

$$W_{as} = 2\sqrt{\gamma_a^{LW}\gamma_s^{LW}} + 2\sqrt{\gamma_a^{+}\gamma_s^{-}} + 2\sqrt{\gamma_s^{+}\gamma_a^{-}} \tag{1}$$

Damp condition:

$$\begin{aligned} W_{asw}^{wet} = {} & 2\gamma_w + 2\sqrt{\gamma_a^{LW}\gamma_s^{LW}} + 2\sqrt{\gamma_a^{+}\gamma_s^{-}} + 2\sqrt{\gamma_s^{+}\gamma_a^{-}} - 2\sqrt{\gamma_a^{LW}\gamma_w^{LW}} \\ & - 2\sqrt{\gamma_w^{LW}\gamma_s^{LW}} - 2\sqrt{\gamma_a^{+}\gamma_w^{-}} - 2\sqrt{\gamma_a^{-}\gamma_s^{+}} - 2\sqrt{\gamma_w^{-}\gamma_s^{+}} - 2\sqrt{\gamma_s^{-}\gamma_w^{+}} \end{aligned} \tag{2}$$

Our research calculated the adhesion work between the mentioned 16 kinds of asphalt binder and 3 kinds of aggregate, shown in Table 9.

It can be seen from Table 9 that in the dry condition, limestone has a larger adhesion work than granite and basalt, which is independent on asphalt. However, in the damp condition, the conclusion is the reverse.

In order to use the parameters coming from surface free energy to evaluate moisture damage resistance of asphalt mixture effectively, Bhasin et al. proposed two parameters ER_1 and ER_2, as Eqs. (3) and (4). Alvarez et al. used these parameters to study the effect of mineral fillers on asphalt–aggregate interfaces, and they demonstrated the HMA mix design can benefit from characterization of fillers and mastics in terms of the SFE and subsequent computation of the energy parameters [15]. They also compared asphalt rubber–aggregate and polymer modified asphalt–aggregate systems in terms of surface free energy and energy indices. They found that in terms of the energy indices computed, the fracture resistance, moisture damage susceptibility, and the wettability of the asphalt over the aggregate of asphalt rubber asphalt–aggregate systems can be comparable to that developed by polymer modified asphalt–aggregate systems [16]. However, further research about the relationship between surface free energy indices and actual moisture damage resistance need to be studied.

$$ER_1 = \left|\frac{W_{as}}{W_{asw}^{wet}}\right| \tag{3}$$

Table 10
Pull-off test results.

Combinations	Aggregate	Dry condition			Damp condition			Failure load loss ratio (%)
		Displacement (mm)	Load (kN)	Rare area ratio (%)	Displacement (mm)	Load (kN)	Rare area ratio (%)	
1# asphalt + limestone	1	0.186	0.986	5	1.746	0.597	90	39.5
	2	0.156	1.244	5	1.020	0.714	90	42.6
	3	0.142	1.132	10	0.690	0.782	80	30.9
	Average	0.161	1.121	6.7	1.152	0.698	86.7	37.7
14# asphalt + Granite	1	0.396	1.336	15	1.278	0.892	100	33.2
	2	1.418	1.171	10	0.594	0.758	100	35.3
	3	1.731	1.154	8	1.019	0.733	90	36.5
	Average	1.182	1.220	11.0	0.964	0.794	96.7	35.0
9# asphalt + basalt	1	0.846	1.122	10	0.794	0.837	85	25.4
	2	0.478	1.346	6	0.581	0.999	80	25.8
	3	1.541	1.462	4	0.654	1.047	80	28.4
	Average	0.955	1.310	6.7	0.676	0.961	81.7	26.5
10# asphalt + limestone	1	0.850	0.815	15	1.061	0.603	95	26.0
	2	0.756	1.642	10	1.628	1.229	90	25.2
	3	0.643	1.038	15	2.850	0.755	92	27.3
	Average	0.750	1.165	13.3	1.846	0.862	92.3	26.2
6# asphalt + limestone	1	0.348	1.042	5	0.106	0.782	80	25.0
	2	0.278	2.242	5	0.387	1.709	70	23.8
	3	0.259	1.364	5	0.168	1.182	75	13.3
	Average	0.295	1.549	5	0.220	1.224	75	20.7
15# asphalt + Granite	1	0.520	1.04	10	0.211	0.812	85	21.9
	2	0.220	1.481	15	0.459	1.260	95	14.9
	3	0.311	2.034	10	0.363	1.518	90	25.4
	Average	0.350	1.518	11.7	0.344	1.197	90	20.7

Fig. 3. The failure state of dry sample.

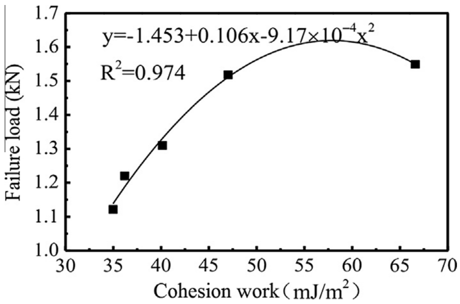

Fig. 4. Relationship between failure load and cohesion work of asphalt.

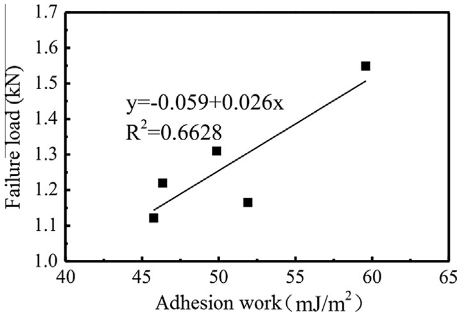

Fig. 5. Relationship between failure load and adhesion work of combination.

$$ER_2 = \left|\frac{W_{as} - W_{aa}}{W_{asw}^{wet}}\right| \tag{4}$$

3.4. Pull-off test

Six combinations were selected to undergo pull-off test to study the effectiveness of ER_1 and ER_2 on evaluating moisture damage resistance of cold mix asphalt (Table 6).

In order to analyze the effect of water on interface between asphalt mastic and aggregate for cold mix asphalt, this section selected two kinds of aggregates to conduct pull-off test. One is dry surface and the other is damp surface. The filler-asphalt mess

Fig. 6. The failure state of damp sample.

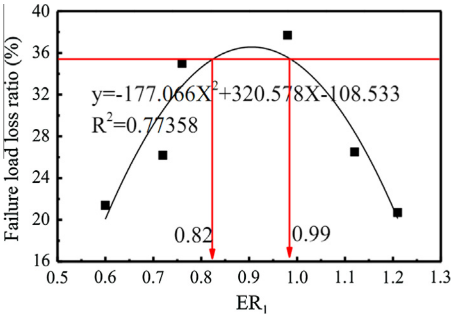

Fig. 7. Relationship between failure load loss ratio and ER_1.

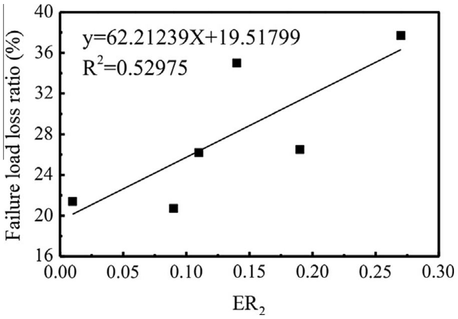

Fig. 8. Relationship between failure load loss ratio and ER_2.

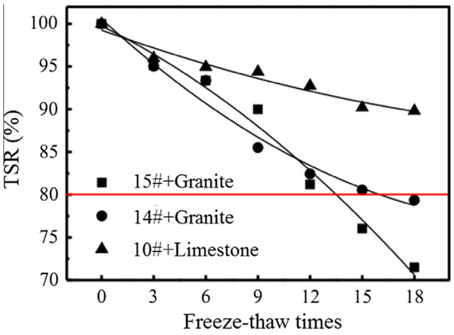

Fig. 9. Effect of freeze–thaw times on tension strength ratio.

ratio of asphalt mastic is 1.1 and the test temperature is −10 °C. The test results are shown in Table 10.

Table 10 shows that the bare area ratio of tested dry sample is between 5% and 15%, which indicates that the failure mainly occurs in the asphalt mastic, not the interface between asphalt mastic and aggregate (Fig. 3). This is generally called cohesion failure. With the increase of adhesion work between asphalt and aggregate, the bare area of failure aggregate surface decreased.

The fit results, shown in Figs. 4 and 5, analyzed the relationship between surface free energy indices and pull-off test results.

Fig. 4 shows that there is a strong quadratic correlation between failure load and cohesion work of asphalt. When the cohesion work increased, the failure load increased. But Fig. 5 shows that there is a weak correlation between failure load and adhesion work between asphalt and aggregate. This demonstrates the failure model between asphalt mastic and dry aggregate is mainly cohesion failure again.

Compared with the dry aggregate, the failure model between damp aggregate and asphalt mastic is adhesion failure and the bare area ratio is more than 70% (Fig. 6). Table 10 also shows that damp aggregate decrease the failure load peak compared to dry aggregate. This is due to the fact that moisture can decrease the adhesion force between asphalt and aggregate.

In order to analyze the relationship between pull-off test and surface free energy indices, we fit the relationship between pull-off failure load loss ratio and ER$_1$, ER$_2$ (Figs. 7 and 8).

Fig. 7 shows that there is a quadratic correlation relationship between pull-off failure load loss ratio and ER$_1$. If the ER$_1$ is less than 0.9, load loss ratio will increase when ER$_1$ increases. However, if the ER$_1$ is higher than 0.9, load loss ratio will decrease when ER$_1$ increases. It can be seen from Fig. 8 that there is a linear positive correlation relationship between failure load loss ratio and ER$_2$, which means that the CMA with a higher ER$_2$ would have a better moisture damage resistance. However, the correlation is weaker than ER$_1$.

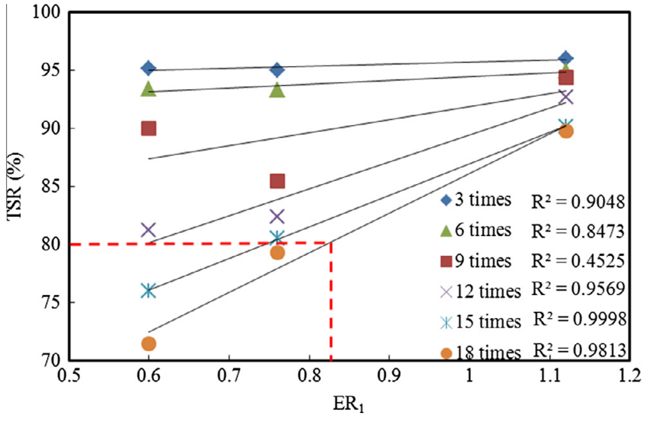

Fig. 10. Relationships between TSR and ER$_1$.

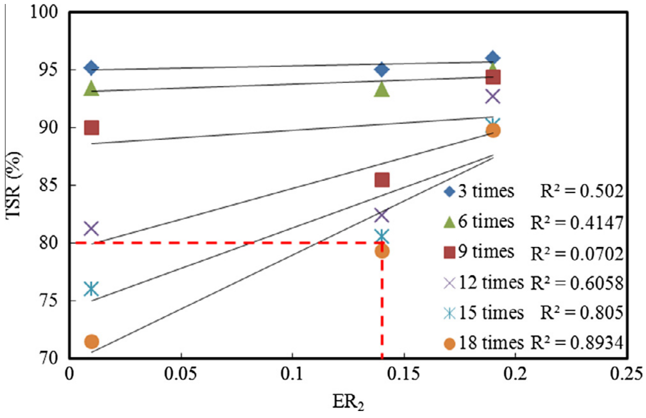

Fig. 11. Relationships between TSR and ER$_2$.

3.5. Freeze–thaw splitting test

First, the effect of freeze–thaw times on tension strength ratio (TSR) was obtained (Fig. 9). The strength of all three kinds of CMA will decrease when freeze–thaw times increase, but the decrease speeds are different. The limestone has a lower decrease speed than granite and basalt.

In order to analyze the correlation between traditional moisture damage evaluation method (TSR) and energy indices, the relationships between TRS and ER$_1$, ER$_2$ were drawn in Figs. 10 and 11.

It can be seen from Fig. 10 that when ER$_1$ increases, TSR increases. This dependence becomes more sensitive when freeze–thaw times increase. This indicates that ER$_1$ is an effective parameter to evaluate the moisture damage resistance of CMA. A larger ER$_1$ represents a better moisture damage resistance. Fig. 10 also shows that the freeze–thaw splitting test becomes effective for evaluating moisture damage resistance only if there are enough freeze–thaw times (our research is 12 times). For our materials in our research, if we want the TSR to be more than 80%, we need ER$_1$ to be at least 0.82. From Fig. 11, we can see that ER$_2$ has a similar conclusion as ER$_1$, and the critical value is 0.14. Using this method, we calculated the energy indices of all combinations (16 asphalt binders × 3 aggregates) in our research. If we define 80% TSR as the critical value, we can clearly see which ones are effective at moisture damage resistance, and which ones are not effective (Figs. 12 and 13). In Figs. 12 and 13, No. 1–No. 16 is asphalt–granite combinations, No. 17–No. 32 is asphalt–basalt combinations, and No. 33–No. 48 is asphalt–limestone combinations.

It can be seen from Figs. 12 and 13 that the fluctuation range of ER$_1$ is between 0.55 and 1.25, while the ER$_2$ is between 0 and 0.35. For the same asphalt binder, moisture damage resistance energy

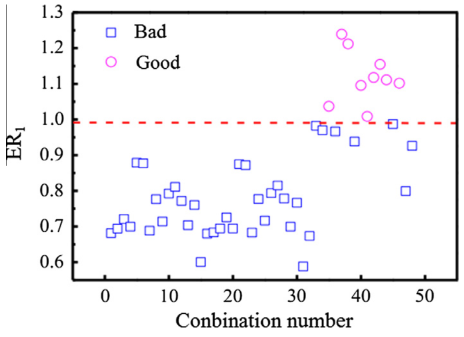

Fig. 12. ER$_1$ of all combinations.

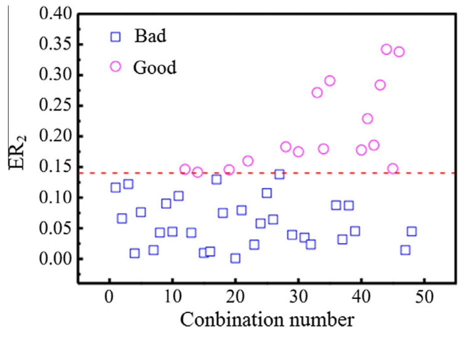

Fig. 13. ER$_2$ of all combinations.

indices are aggregate dependent. For the same aggregate, damage resistance energy indices ARE also dependent on asphalt types. Generally, limestone has a better moisture damage resistance.

4. Conclusions

Based on the testing and analysis presented herein, the conclusions of the study are summarized as follows:

- Diluents and additives used in cold mix asphalt can increase surface free energy of pure asphalt. The significant rank of the factors effecting SFE is diluent types > additives content > diluent content > additives types. SFE of aggregates used in our research are of the following rank: Limestone > Basalt > Granite.
- In dry conditions, compared with basalt and granite, the adhesion work between limestone and all asphalts are the maximum, while in damp conditions, they become the minimum.
- Pull-off test demonstrates that the cohesion failure usually occurs in dry conditions, while adhesion failure usually occurs in damp conditions. There is a quadratic correlation relationship between pull-off failure load loss ratio and energy indices ER_1, and a linear positive correlation relationship between failure load loss ratio and energy indices ER_2.
- The strength of all CMAs will decrease when freeze–thaw times increase. There is a strong linear relationship between TSR and ER_1 (or ER_2), which indicates that energy indices are a good method to quantify the moisture damage resistance of CMA. Limestone is generally better at resisting moisture damage than granite and basalt.

Acknowledgments

We are grateful for financial support from China Scholarship Council (Grant number: 201306120187) and National Science Fund for Distinguished Young Scholars of China (Grant number: 51225803).

References

[1] Munyagi Anna Abela. Evaluation of cold asphalt patching mixes. Stellenbosch. M.S. Dissertation. University of Stellenbosch; 2006.

[2] Chavez-Valencia LE, Alonso E, Manzano A, Perez J, Contreras ME, Signoret C. Improving the compressive strengths of cold-mix asphalt using asphalt emulsion modified by polyvinyl acetate. Constr Build Mater 2007;21:583–9.

[3] Benedito de S Bueno, da Silva Wander R, de Lima Dario C, Minete Enivaldo. Engineering properties of fiber reinforced cold asphalt mixes. J Environ Eng 2003;129:952–5.

[4] Al-Busaltan Shakir, Al Nageim Hassan, Atherton William, Sharples George. Mechanical properties of an upgrading cold-mix asphalt using waste materials. J Mater Civil Eng 2012;24(12):1484–91.

[5] Serfass JP. Advantages peculiarities and mysteries of emulsion cold mixes. Rev Gener Routes Autoroutes 2002;802:60–8.

[6] Khalid Hussain A, Monney Ormsby K. Moisture damage potential of cold asphalt. Int J Pavement Eng 2009;10(5):311–8.

[7] Doyle Thomas A, McNally Ciaran, Gibney Amanda, Tabakovic Amir. Developing maturity methods for the assessment of cold-mix bituminous materials. Constr Build Mater 2013;38:524–9.

[8] Cheng D. Surface free energy of asphalt–aggregate system and performance analysis of asphalt concrete based on surface free energy. College Station: Texas A&M University; 2002.

[9] Bhasin A, Howson JE, Masad E, Little D, Lytton RL. Effect of modification processes on bond energy of asphalt binders. Transp Res Rec 2007; 1988:29–37.

[10] Little DN, Bhasin A. Using surface energy measurements to select materials for asphalt pavement. Texas transportation institute, final report of project 9–37, Washington (DC): National Cooperative Highway Research, Program; 2006.

[11] Tan Yiqiu, Guo Meng. Interfacial thickness and interaction between asphalt and mineral fillers. Mater Struct 2014;47:605–14.

[12] Tan Yiqiu, Guo Meng. Using surface free energy method to study the cohesion and adhesion of asphalt mastic. Constr Build Mater 2013;47:254–60.

[13] Bhasin A, Little DN, Vasconcelos KL, Masad E. Surface free energy to identify moisture sensitivity of materials for asphalt mixes. Transp Res Rec 2007; 2001:37–45.

[14] Wei JM, Zhang YZ. Application of sessile drop method to determine surface free energy of asphalt and aggregate. J Test Eval 2012;40(5):807–13.

[15] Alvarez Allex E, Ovalles Evelyn, Caro Silvia. Assessment of the effect of mineral filler on asphalt–aggregate interfaces based on thermodynamic properties. Constr Build Mater 2012;28:599–606.

[16] Alvarez Allex E, Ovalles Evelyn, Epps Martin Amy. Comparison of asphalt rubber–aggregate and polymer modified asphalt–aggregate systems in terms of surface free energy and energy indices. Constr Build Mater 2012;35:385–92.