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Research Article

Preparation Parameter Analysis and Optimization of Sustainable Asphalt Binder Modified by Waste Rubber and Diatomite

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In this study, crumb rubber and diatomite were used to modify asphalt binder. Wet process was adopted as a preparation method, and the corresponding preparation process was determined firstly. The effects of six preparation parameters (crumb rubber concentration, diatomite concentration, shear time, shear speed, shear temperature, and storing time) on properties of modified asphalt binder (penetration at 25°C, softening point, ductility, viscosity at 135°C, elastic recovery, and penetration index) were investigated, and multiresponse optimization was conducted using the response surface method. The results revealed that softening points, viscosity, elastic recovery, and penetration index increase, while penetration and ductility decrease with the increase of crumb rubber concentration. Softening points, viscosity, and penetration index increase, while penetration and ductility decrease with the increase of diatomite concentration, which presents little influence on elastic recovery of binder. Shear temperature presented significant effects on penetration, softening point, viscosity, and ductility. Shear speed, shear time, and storing time have similar effects on binder properties because of their similar mechanism of action. Based on the model obtained from the response surface method, optimized preparation parameters corresponding to specific criteria can be determined, which possess favorable accuracy compared with experimental results.

1. Introduction

Disposal of industrial wastes has become a critical problem in the world under the promotion of environmental management and mission of sustainable development [1, 2]. Millions of end-of-life tyres (ELTs) are generated each year due to the increasing number of vehicles, which have been regarded as one of the largest and most problematic waste polymeric materials with large quantity and durability [3]. Current researches have shown that crumb rubber (CR) derived from grinding of ELTs into asphalt modification can be an effective modifier for construction and maintenance of asphalt pavement [4]. For one thing, CR-modified asphalt and corresponding mixture possess favorable environmental benefits. Farina et al. [5] demonstrated the environmental results of recycled materials in bituminous mixtures for pavement wearing courses using life cycle assessment (LCA) methodology. Results reveal that the use of wearing course containing asphalt rubber obtained by the wet process presents significant benefits for energy saving, environmental impact, human

health, preservation of ecosystems, and minimization of resource depletion. The reductions of the gross energy requirement and global warming potential range between 36% and 45% compared with standard paving solutions. Bartolozzi et al. [6] compared the environmental performances between rubberized asphalt pavement and the conventional one. Results show that the global environmental performances of the rubberized asphalt road are improved by 30–40%. La Rosa et al. [7] evaluated the environmental benefit of using recycled waste tyres in mixture with virgin rubber. It can be concluded that addition of ground tyre rubber (GTR) in styreneisoprene-styrene (SIS) formulation considerably reduces the influences in terms of global warming potential, human toxicity, energy consumption, and so on. Yu et al. [8] evaluated the environmental burdens of plastic-rubber asphalt (PRA) mixture and styrene-butadiene-styrene (SBS) asphalt mixture by using a cradle-to-gate LCA model. The results revealed that PRA mixture is more environmental-friendly than SBS asphalt mixture. CR has been proved to be an effective modifier for virgin asphalt. Adding CR into asphalt

binder can improve its viscosity and elastic properties at high temperature, which can enhance the resistance ability to permanent deformation under traffic load. It can also increase the flexibility of asphalt binder at low temperature, thus making it more capable of resisting low temperature cracking [9–13]. Therefore, recycled ELTs have been widely used as a road-paving material in the world.

Diatomite is a naturally occurring, soft, siliceous sedimentary rock that can be easily crumbled into fine powder. The typical chemical components of diatomite are silica, alumina, and iron oxide [14]. Diatomite has been widely used as a asphalt modifier due to its low cost, large storage, and high absorptive ability [14-18]. The addition of diatomite into asphalt can improve its high temperature performance, storage stability, rutting resistance, and longterm aging resistance. Asphalt mastic modified by diatomite possesses more favorable high and medium temperature performances than that modified by limestone, hydrated lime, and fly ash [16]. However, diatomite-modified asphalt presents some drawbacks such as reducing the thermal conductivity and low temperature deformation ability of asphalt mixture [17]. In order to overcome this limitation, crumb rubber and diatomite can be used as a compound modifier for asphalt binder. Liu et al. [19] investigated the short-term aging properties of diatomite and crumb rubber compound-modified asphalt (DRA). The results revealed that DRA could combine the advantages of diatomite and crumb rubber and achieve better performance in short-term aging resistance than diatomite-modified asphalt (DA) and crumb rubber-modified asphalt (RA).

It is worth noting that different procedures and parameters can be applied to prepare crumb rubber and diatomite compound-modified asphalt binders [20]. The different preparation procedures and parameters can result in different interactions among crumb rubber, asphalt, and diatomite, thus affecting the properties of modified asphalt. But previous studies have rarely investigated the effect of preparation factors on the property of crumb rubber and diatomite compound-modified asphalt, and even the influences of preparation factors on properties of crumb rubber-modified asphalt and diatomite-modified asphalt are limited [20, 21].

In general, there are two kinds of production methods for crumb rubber-modified asphalt binder: the dry process and wet process [3, 22]. In the dry process, coarse crumb rubber is used to substitute a part of the mineral aggregate, which is suitable for producing asphalt mixture with favorable elasticity and anti-icing properties. In the wet process, crumb rubber and asphalt are mixed and allowed to react for a period that can take advantage of the benefits of both base ingredients. Crumb rubber-modified asphalt obtained through the wet process has spread worldwide and got much success by roads built in the past 30 years [3]. For the production of diatomite-modified asphalt, the wet process is the most widely used method. In the preparation of both crumb rubber-modified asphalt binder and diatomite-modified one, the device of high-speed mixing and shear stirring is usually adopted to obtain a homogeneous asphalt binder. The processing parameters including temperature, time, and speed determine the interaction

condition, which is important for the properties of modified asphalt [23]. However, different preparation parameters were applied in recent researches. For the preparation of crumb rubber-modified asphalt, shear temperature 175°C, shear speed 5000 rpm, and shear time 40 min were adopted by Wang et al. [24]. Shear temperature 170-180°C, shear speed 5000 rpm, and shear time 5 min were used by Kedarisetty et al. [23]. In the research by Peralta et al. [25], asphalt rubber binder was heated at 180°C for 60 min and stirred at 230 rpm. For the preparation of diatomitemodified asphalt, the shear temperature is 150°C, shear speed is 3000 rpm, and shear time is 120 min in the research by Cong et al. [14]. The shear temperature is 150°C, shear speed is 4000 rpm, and shear time is 40 min in the research by Guo et al. [17]. The shear temperature is 160°C, shear speed is 5000 rpm, and shear time is 40 min in the research by Tan et al. [15]. As can be concluded from current researches, different preparation parameters were adopted. The effects of preparation parameters on properties of modified asphalt have not been demonstrated.

In this study, the preparation process for crumb rubber and diatomite compound–modified asphalt binder was determined. The effects of six kinds of preparation parameters (crumb rubber concentration, diatomite concentration, shear time, shear speed, shear temperature, and storing time) on properties of binder (penetration at 25°C, softening point, ductility, viscosity at 135°C, elastic recovery, and penetration index (PI)) were investigated using the response surface method (RSM). Relationships between preparation parameters and asphalt properties were obtained, and multiresponse optimization was conducted to determine the optimum preparation parameters corresponding to the satisfactory properties.

2. Materials and Methods

2.1. Raw Materials. Base asphalt AH-90 from Panjin Petrochemical Industry, crumb rubber particle from Changchun Yuxing Rubber Materials Co., Ltd., and diatomite produced by Changchun Diatomite Products Co., Ltd. were used in this study. The detailed properties for raw materials were given in [19].

2.2. Preparation of Crumb Rubber and Diatomite Compound-Modified Asphalt. The wet process was adopted to prepare the crumb rubber and diatomite compound-modified asphalt. A high-speed shear homogenizer (KRH-I, Shanghai Konmix Mechanical & Electrical Equipment Technology Co. Ltd., China) was employed, which can control its internal temperature through a heating device and oil bath. The preparation process mainly included three steps: heating of raw materials; shearing and mixing of asphalt, crumb rubber, and diatomite; and storing of modified asphalt binder. In this study, the influences of six important preparation parameters on compound-modified asphalt binder were investigated. They were content of crumb rubber by weight of the neat asphalt, shear temperature,

shear speed, shear time, and storing time. Contents of crumb rubber and diatomite are essential parameters affecting the properties of compound-modified asphalt. Shear speed, shear time, shear temperature, and storing time are key factors in the interaction process among asphalt, crumb rubber, and diatomite to achieve a homogeneous and stable modified binder. The adopted six preparation parameters have not been standardized in current researches, and the researchers have used various parameters. Based on previous research results and considering the workability of binder, the range for content of crumb rubber was 5-15%; it was 5-15% for content of diatomite, 160-190°C for shear temperature, 3000-6000 rpm for shear speed, 30-60 min for shear time, and 30-60 min for storing time in this study. Therefore, the detailed preparation process was determined as follows: firstly, neat asphalt, crumb rubber, and diatomite were separately heated in an oven at 140°C, and asphalt was at the fluid state that can be easily stirred. Secondly, the crumb rubber and diatomite were added into asphalt, which was placed into a shear homogenizer after preliminary mixing. Then, shear temperature and speed were set to the specific values; the blend of asphalt, crumb rubber, and diatomite was mixed and sheared for the specified shear time after the temperature of the blend reached the specified shear temperature. Finally, the blend was contained in the shear homogenizer to swell for the specified storing time. The crumb rubber and diatomite compound-modified asphalt binder was prepared for the tests.

2.3. Characterization Method. In order to evaluate the effects of content of crumb rubber, content of diatomite, shear temperature, shear speed, shear time, and storing time on asphalt properties, penetration, softening point, ductility, viscosity, elastic recovery, and penetration index (PI) were tested and investigated for compound-modified asphalt binder. Detailed illustrations for penetration, softening point, ductility, viscosity, and elastic recovery were conducted in [19].

The results obtained from penetration and softening point tests can be used to calculate the penetration index (PI). PI is an important property which is related to Van der Poel's monograph and useful to determine the stiffness of asphalt. PI was usually used for the classification of pure asphalt. However, it has been verified to be useful for polymer-modified ones [26]. PI can be calculated by the following classical approach [27]:

PI =
$$\frac{30}{1 + 50 \left(\lg 800 - \lg P_{(25^{\circ}C, 100 \text{ g.5 s})} / T_{\text{R&B}} - 25 \right)} - 10,$$
(1)

where $P_{(25^{\circ}\text{C},100\text{ g},5\text{ s})}$ is the penetration at 25°C and $T_{\text{R\&B}}$ is the softening point temperature.

2.4. Response Surface Method. In order to analyze the effects of six preparation parameters (content of crumb rubber, content of diatomite, shear temperature, shear speed, shear time, and storing time) on the asphalt property, the response

surface method (RSM) was utilized. The RSM is a group of mathematical and statistical techniques useful to design experiments. It can save cost and time by reducing the overall number of tests required [28, 29]. The RSM has been widely used in many areas to identify the effect of individual variables and interaction of different independent variables on the response [30–32]. An optimal quadratic model was used to determine the optimal condition of response:

$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i \le i}^k \beta_{ij} x_i x_j + \varepsilon,$$
 (2)

where y is the predicted response; x_i and x_j are the coded values of the preparation parameters; k is the number of independent variables; β_i is the linear effect of x_i ; β_{ij} is the linear interaction between x_i and x_j ; β_{ii} is the secondary effects of x_i ; and ε is the random error.

In this study, the three level Box-Behnken design with six factors (content of crumb rubber, X_1 ; content of diatomite, X_2 ; shear time, X_3 ; shear speed, X_4 ; shear temperature, X_5 ; and storing time, X_6) was carried out. Actual and coded values of the independent variables are shown in Table 1. Combinations of X_1 (5%, 10%, 15%), X_2 (5%, 10%, 15%), X_3 (30, 45, 60 min), X_4 (3000, 4500, 6000 rpm), X_5 (160°C, 175°C, 190°C), and X_6 (30, 45, 60 min) were selected as independent variables. Penetration at 25°C, Y_1 ; softening point, Y_2 ; ductility at 15°C, Y_3 ; viscosity at 135°C, Y_4 ; elastic recovery at 25°C, Y_5 ; and penetration index (PI), Y_6 were adopted as the responses. The software Design-Expert 7.0 was adopted for the scheme design, model generating, statistical analysis, and optimization of the preparation parameters.

3. Results and Discussion

3.1. Experimental Scheme and ANOVA Results

3.1.1. Experimental Scheme. Experimental scheme for property analysis of compound-modified asphalt binder under the effect of preparation parameters was established based on the RSM. This scheme is composed of 54 experimental groups including 6 replicates at the central point, which is much less than a full test plan $3^6 = 729$. Three specimens are included in each group, and the corresponding average value is regarded as a representative one. The detailed experimental scheme and test results of responses are listed in Table 2. The relationships between independent variables and responses were analyzed by 3D response surface plots. A 3D plot is used to represent the dependent variables in function of two independent parameters, when other four variables are kept constant at level 0 in the code value. The interaction between independent variables can also be demonstrated in a 3D plot.

As can be seen from Table 2, the penetration ranged from 34.8 to 71.0 (0.1 mm). Softening point ranged from 46.8°C to 62.4°C. Ductility ranged from 141.0 to 379.7 mm. Viscosity ranged from 497.3 to 5752.0 mPa·s. Elastic recovery ranged from 21% to 63%. PI ranged from -1.5 to 0.83. According to these experimental ranges, the minimum and maximum values of penetration were obtained at Group

V (:)		Actual value								
X_6 (min)	X_5 (°C)	X_4 (rpm)	X_3 (min)	X_{2} (%)	X_1 (%)	Coded value				
30	160	3000	30	5	5	-1				
45	175	4500	45	10	10	0				
60	190	6000	60	15	15	+1				
_	175	4500	45	10 15	1.5	-1 0 +1				

TABLE 1: Actual and coded values of the independent variables.

nos. 50 (X_1 : 15%; X_2 : 15%; X_3 : 45 min; X_4 : 3000 rpm; X_5 : 175°C; and X_6 : 45 min) and 46 (X_1 : 5%; X_2 : 5%; X_3 : 45 min; X_4 : 3000 rpm; X_5 : 175°C; and X_6 : 45 min), respectively. The minimum and maximum values of softening point were obtained at Group nos. 46 (X_1 : 5%; X_2 : 5%; X_3 : 45 min; X_4 : 3000 rpm; X_5 : 175°C; and X_6 : 45 min) and 50 (X_1 : 15%; X_2 : 15%; X_3 : 45 min; X_4 : 3000 rpm; X_5 : 175°C; and X_6 : 45 min), respectively. The minimum and maximum values of ductility were obtained at Group nos. 54 (X_1 : 15%; X_2 : 10%; X_3 : 45 min; X_4 : 3000 rpm; X_5 : 160°C; and X_6 : 45 min) and 14 (X_1 : 15%; X_2 : 5%; X_3 : 45 min; X_4 : 4500 rpm; X_5 : 190°C; and X_6 : 60 min), respectively. The minimum and maximum values of viscosity were obtained at Group nos. 46 $(X_1: 5\%; X_2: 5\%; X_3: 45 \text{ min}; X_4: 3000 \text{ rpm}; X_5: 175^{\circ}\text{C}; \text{ and}$ X_6 : 45 min) and 50 (X_1 : 15%; X_2 : 15%; X_3 : 45 min; X_4 : 3000 rpm; X_5 : 175°C; and X_6 : 45 min), respectively. The minimum and maximum values of elastic recovery were obtained at Group nos. 21 (X_1 : 10%; X_2 : 10%; X_3 : 45 min; X_4 : 4500 rpm; X_5 : 190°C; and X_6 : 60 min) and 50 (X_1 : 15%; X_2 : 15%; X_3 : 45 min; X_4 : 3000 rpm; X_5 : 175°C; and X_6 : 45 min), respectively. The minimum and maximum values of PI were obtained at Group nos. 49 (X_1 : 15%; X_2 : 5%; X_3 : 45 min; X_4 : 3000 rpm; X_5 : 175°C; and X_6 : 45 min) and 7 (X_1 : 5%; X_2 : 10%; X_3 : 30 min; X_4 : 4500 rpm; X_5 : 175°C; and X_6 : 60 min), respectively.

3.1.2. Analysis of Variance (ANOVA) Results for Quadratic Models and Independent Variables. Analysis of variance (ANOVA) was used to evaluate the statistical significance of independent parameters and interactions among them. The adequacy of the models constructed for penetration at 25°C, softening point, ductility at 15°C, viscosity at 135°C, elastic recovery at 25°C, and penetration index (PI) was checked by coefficient of determination (R^2), adjusted coefficient of determination (Adj. R^2), Adeq. precision, and Fisher's test value (F value). Models and factors were considered significant when p < 0.01. ANOVA results for quadratic models and independent variables are obtained and listed in Tables 3 and 4, respectively. The detailed explanations of results are given in Sections 3.2–3.7.

3.2. Penetration

3.2.1. Analysis of the Model for Penetration. The results of ANOVA for the penetration model indicated that the model possessed satisfactory levels of R^2 (0.90), Adj. R^2 (0.80), and Adeq. precision (11.014), which were significant at p < 0.0001. R^2 and Adj. R^2 values of the model were higher than 0.8, which revealed a close agreement between experimental results and predicted ones. Adeq. precision is the measure

of the signal-to-noise ratio, and greater ratio than 4 is desirable. An Adeq. precision of 11.014 indicated that the model can be used to navigate the design space.

The second-order quadratic polynomial models were established based on the regression coefficients that were determined with the least squares method. Stepwise regression was used to identify the statistically significant variables. As can be seen from the ANOVA results of independent variables for the penetration model, the significant variables (p < 0.05) for the penetration model included $X_1, X_2, X_5, X_{23}, X_{46}, X_{22}, X_{33}$, and X_{44} . The reduced second-order model in terms of coded factors for penetration was obtained by removing the insignificant variables:

$$\begin{split} Y_{1} &= 43.81 - 6.69 \times X_{1} - 4.97 \times X_{2} + 5.17 \times X_{5} \\ &+ 3.10 \times X_{23} - 3.01 \times X_{46} + 2.87 \times X_{22} + 6.24 \times X_{33} \\ &+ 6.22 \times X_{44}. \end{split} \tag{3}$$

3.2.2. Effect of Preparation Parameters on Penetration. As shown in Table 4, the most significant linear variables on penetration include content of crumb rubber, content of diatomite, and shear temperature with p < 0.0001. Meanwhile, quadratic terms of shear time and shear speed present the most significant effect with p < 0.0001. The quadratic term of diatomite content has a significant effect with p < 0.05. Furthermore, the interaction terms of (diatomite content) * (shear time) and (shear speed) * (storing time) have significant effects on penetration with p < 0.05.

The relationships between preparation parameters and penetration were demonstrated in 3D response surface plots, which are shown in Figure 1. As can be seen from the figure, the penetration decreases with the increase of diatomite concentration and crumb rubber concentration. However, it increases with the increase of shear temperature. For the relationships of penetration with shear time, shear speed, and storing time, they present the trends of decreasing firstly and then increasing.

The interaction of crumb rubber and asphalt binder is essential for the performance of modified asphalt binder. Rubber particle swelling and significant absorption take place when rubber particles are added into heated asphalt binder. The polymer chains of rubber particles can absorb the lighter and solvating fractions of binder. Rubber particle size and shape are changed, and a gel-like structure is generated [3], which causes the reduction of penetration for asphalt binder.

Diatomite has a large specific area and good adsorption performance, which can effectively absorb the lower molecular group and lower polar aromatic molecular group for its mesoporous structure. The anchorage structure forms

Table 2: Experimental scheme with test results for property analysis of compound-modified asphalt binder.

Group				n paramete			try unaryons or		Respon			
	X_1 (%)	X_{2} (%)	-	•		X_6 (min)	Y_1 (0.1 mm)	Y ₂ (°C)	-		Y_{5} (%)	Y_6
1	5	10	45	6000	160	45	47.9	52.5	185.0	1343.3	33	-0.69
2	15	10	30	4500	175	30	47.5	56.2	218.3	2682.3	49	0.12
3	10	5	30	4500	190	45	67.7	50.5	339.5	1280.0	36	-0.33
4	15	5	45	6000	175	45	51.0	57.2	259.0	2865.0	58	0.50
5	10	15	45	4500	160	30	42.6	55.4	158.3	2098.0	36	-0.31
6	10	10	45	4500	175	45	44.8	53.3	203.3	1907.0	43	-0.66
7	5	10	30	4500	175	60	60.8	47.1	295.0	692.4	25	-1.50
8	10	10	45	4500	175	45	42.4	54.5	205.6	1919.0	44	-0.52
9	10	15	30	4500	190	45	50.6	54.8	294.3	1792.7	42	-0.04
10 11	10 15	10 10	45 30	4500 4500	175 175	45 60	44.3 51.4	53.9 55.1	203.3 244.0	1907.0 1778.7	43 44	-0.55 0.07
12	10	10	45	4500	175	45	43.6	53.1	204.5	1906.0	44	-0.58
13	10	5	45	4500	160	60	45.3	53.1	181.5	1833.3	42	-0.58
14	10	5	45	4500	190	60	55.0	51.0	379.7	1676.0	41	-0.73
15	10	10	30	3000	175	30	55.5	54.1	215.5	1844.7	34	0.03
16	5	10	30	4500	175	30	54.6	51.2	255.7	1140.7	25	-0.70
17	10	10	45	4500	175	45	43.1	54.1	204.9	1906.5	44	-0.57
18	10	15	60	4500	190	45	56.9	55.3	197.0	2017.0	51	0.36
19	10	5	30	4500	160	45	53.1	53.6	175.0	1402.0	40	-0.20
20	10	15	30	4500	160	45	41.8	55.9	171.0	2513.0	36	-0.24
21	10	10	30	3000	175	60	58.8	51.8	316.5	1301.7	21	-0.37
22	10	10	45	4500	175	45	44.8	53.3	203.3	1907.0	43	-0.66
23	10	5	60	4500	190	45	61.6	50.5	301.0	953.1	41	-0.58
24	5	5	45	6000	175	45	68.6	50.6	284.0	770.1	31	-0.27
25	15	10	45	6000	160	45	36.9	59.6	172.7	3821.0	54	0.24
26	10	5 10	60 45	4500	160	45 45	47.8	52.7	184.0	1034.0 880.4	46	-0.65
27 28	5 10	10	45 30	3000 6000	160 175	45 60	54.6 53.8	50.7 54.2	219.3 231.0	2008.3	27 38	-0.82 -0.02
29	10	15	45	4500	190	60	53.1	55.4	243.5	2109.0	53	0.21
30	15	10	60	4500	175	30	43.6	59.2	214.3	3675.7	57	0.54
31	5	15	45	6000	175	45	48.8	53.2	216.3	1356.0	29	-0.50
32	10	5	45	4500	190	30	56.8	51.4	341.3	1627.0	42	-0.55
33	10	10	60	6000	175	60	56.6	52.4	308.5	1930.3	47	-0.32
34	10	10	30	6000	175	30	57.9	52.2	354.3	1508.0	33	-0.31
35	5	10	45	3000	190	45	63.0	49.4	315.0	1188.0	32	-0.80
36	10	10	60	3000	175	30	53.3	54.0	269.5	2229.7	44	-0.09
37	10	15	60	4500	160	45	49.0	56.0	174.0	2430.0	42	0.15
38	10	15	45	4500	190	30	49.7	54.5	260.3	2054.0	46	-0.15
39	5	10	60	4500	175	60	49.9	50.9	249.0	913.4	34	-0.99
40	10	5	45	4500	160	30	49.2	53.3	197.7	1860.0	41	-0.44
41	5	15	45	3000	175	45	55.2	51.5	357.7	1202.7	15	-0.60
42 43	10 5	10 10	60 45	3000 6000	175 190	60 45	60.3 59.3	50.9 49.9	329.0 314.5	774.4 704.8	25 30	-0.53 -0.82
43 44	5 15	15	45 45	6000	175	45 45	39.3 37.1	60.4	314.5 191.7	704.8 4847.3	61	-0.82 0.40
45	10	10	60	6000	175	30	66.3	49.5	348.0	1735.0	41	-0.65
46	5	5	45	3000	175	45	71.0	46.8	307.3	497.3	23	-0.03
47	15	10	45	3000	190	45	50.6	57.6	292.3	3509.0	54	0.57
48	15	10	45	6000	190	45	47.0	57.0	278.3	3539.0	60	0.26
49	15	5	45	3000	175	45	50.8	58.8	224.5	3724.3	55	0.83
50	15	15	45	3000	175	45	34.8	62.4	160.0	5752.0	63	0.64
51	10	15	45	4500	160	60	39.1	55.6	170.0	2198.7	32	-0.45
52	5	10	60	4500	175	30	58.4	49.1	309.5	765.9	23	-1.07
53	15	10	60	4500	175	60	40.6	59.5	198.7	4491.7	59	0.43
54	15	10	45	3000	160	45	40.2	59.3	141.0	3916.3	45	0.37

and the stiffening effect presents [33], which decrease the penetration of binder.

As for shear temperature, the interactions among asphalt, crumb rubber, and diatomite are strengthened with the

increase of shear temperature. Rubber particle dissolution happens because of depolymerization and devulcanization, which break not only the polymer chains of rubber but also the crosslink bonds. Then, the swelling reaction is reduced,

Sources	R^2	Adj. R ²	Adeq. precision	F value	p value	Significant
Y_1	0.90	0.80	11.014	8.68	< 0.0001	Yes
Y_2	0.95	0.90	17.854	18.37	< 0.0001	Yes
Y_3	0.90	0.79	10.875	8.46	< 0.0001	Yes
Y_4	0.94	0.87	13.392	13.94	< 0.0001	Yes
Y_5	0.92	0.84	14.374	11.01	< 0.0001	Yes
Y_6	0.93	0.85	14.466	12.00	< 0.0001	Yes

TABLE 3: ANOVA results for quadratic models of modified asphalt properties.

TABLE 4: ANOVA results for independent variables.

Sources		Y_1		Y_2		Y_3		Y_4		Y_5		Y_6
Sources	F	p	F	p	F	Р	F	Р	F	Р	F	Р
X_1	74.86	<0.0001****	358.54	<0.0001****	26.1	<0.0001****	220.32	<0.0001****	279.97	<0.0001****	236.0	<0.0001****
X_2	41.34	<0.0001****	60.43	< 0.0001 ****	17.3	0.0003***	23.60	<0.0001****	0.19	0.6701	15.2	0.0006***
X_3	0.26	0.6144	0.38	0.5416	0.0	0.8445	1.81	0.1899	19.69	0.0001****	0.0	0.9208
X_4	0.84	0.3668	0.07	0.7993	0.0	0.9755	0.03	0.8620	15.23	0.0006***	0.0	0.8415
X_5	44.66	< 0.0001 ****	15.07	0.0006***	104.4	<0.0001****	1.66	0.2085	6.66	0.0159*	1.4	0.2500
X_6	0.33	0.5691	0.36	0.5541	0.0	0.9802	0.46	0.5041	0.24	0.6299	1.8	0.1901
X_{12}	0.27	0.6055	0.02	0.8832	2.0	0.1675	4.45	0.0448*	2.78	0.1077	1.4	0.2395
X_{13}	0.49	0.4923	3.59	0.0692	0.5	0.4847	8.96	0.0060**	2.33	0.1388	1.3	0.2606
X_{14}	0.98	0.3306	7.37	0.0116*	6.2	0.0197**	1.50	0.2312	0.35	0.5609	7.2	0.0125*
X_{15}	0.00	0.9454	0.02	0.8961	0.2	0.6962	0.08	0.7832	1.16	0.2905	0.4	0.5503
X_{16}	0.09	0.7717	0.26	0.6137	0.1	0.7023	0.03	0.8700	1.59	0.2184	1.1	0.3138
X_{23}	5.37	0.0286*	0.24	0.6252	0.6	0.4287	0.42	0.5223	0.14	0.7099	7.0	0.0134*
X_{24}	0.03	0.8562	0.71	0.4083	2.2	0.1461	0.02	0.8992	0.00	0.9543	1.8	0.1916
X_{25}	0.27	0.6110	2.23	0.1478	7.0	0.0137**	0.14	0.7147	11.70	0.0021**	3.4	0.0758
X_{26}	0.28	0.6017	0.31	0.5800	0.1	0.7374	0.01	0.9184	0.07	0.7989	1.3	0.2724
X_{34}	1.23	0.2783	1.33	0.2591	0.0	0.9543	0.05	0.8231	0.11	0.7442	0.4	0.5388
X_{35}	0.03	0.8635	0.18	0.6718	3.4	0.0782	0.07	0.7891	0.02	0.8861	0.2	0.6993
X_{36}	2.40	0.1332	2.93	0.0986	0.8	0.3941	0.36	0.5522	0.48	0.4967	1.1	0.3038
X_{45}	0.07	0.8001	0.53	0.4748	0.0	0.8845	0.41	0.5299	0.68	0.4156	0.3	0.5728
X_{46}	5.05	0.0334*	11.53	0.0022**	16.1	0.0005***	4.37	0.0466*	12.78	0.0014**	6.7	0.0158*
X_{56}	0.70	0.4095	0.03	0.8704	0.1	0.7497	0.00	0.9816	0.46	0.5027	1.0	0.3236
X_{11}	0.39	0.5398	7.87	0.0094**	0.4	0.5287	10.11	0.0038**	0.84	0.3692	3.6	0.0693
X_{22}	5.90	0.0223*	2.61	0.1183	0.9	0.3585	1.37	0.2530	0.10	0.7563	13.0	0.0013**
X_{33}	27.96	<0.0001****	3.72	0.0647	4.3	0.0471*	4.40	0.0458*	3.92	0.0585	5.1	0.0318*
X_{44}	27.76	<0.0001****	0.38	0.5442	23.2	<0.0001****	0.50	0.4840	5.90	0.0224*	13.0	0.0013**
X_{55}	0.29	0.5974	0.04	0.8452	0.0	0.8253	0.47	0.4968	0.09	0.7687	0.3	0.5725
X_{66}	1.71	0.2025	3.17	0.0869	11.8	0.0020**	0.10	0.7596	3.99	0.0562	0.8	0.3675

Note. * $p \le 0.05$, ** $p \le 0.01$, *** $p \le 0.001$, and **** $p \le 0.0001$.

and the lighter fractions of binder increase. Therefore, the penetration increases with the increase of shear temperature.

Shear speed, shear time, and storing time present similar effects on penetration. At the early stage, rubber and diatomite particles are distributed more evenly in asphalt binder with the increase of shear speed, shear time, and storing time. The swelling effect is significant, and distances between particles are shortened. A gel-like structure and an anchorage structure are formed and strengthened. The penetration decreases. However, the crosslink bonds between particles are destroyed if the shear time and storing time are longer and the shear speed becomes higher, which increase the penetration of binder.

3.3. Softening Point

3.3.1. Analysis of the Model for Softening Point. The softening point model possessed satisfactory levels of R^2 (0.95),

Adj. R^2 (0.90), and Adeq. precision (17.854), which were significant at p < 0.0001. R^2 and Adj. R^2 values of the model were higher than 0.8, which revealed a close agreement between experimental results and predicted ones. An Adeq. precision of 17.854 indicated that the model can be used to navigate the design space.

As can be seen from the ANOVA results of independent variables for the softening point model, the significant variables (p < 0.05) included $X_1, X_2, X_5, X_{14}, X_{46}$, and X_{11} . The reduced second-order model in terms of coded factors for softening point was obtained by removing the insignificant variables:

$$\begin{split} Y_2 &= 53.83 + 4.15 \times X_1 + 1.70 \times X_2 - 0.85 \times X_5 \\ &- 0.73 \times X_{14} + 1.29 \times X_{46} + 0.94 \times X_{11}. \end{split} \tag{4}$$

3.3.2. Effect of Preparation Parameters on Softening Point. As shown in Table 4, the most significant linear

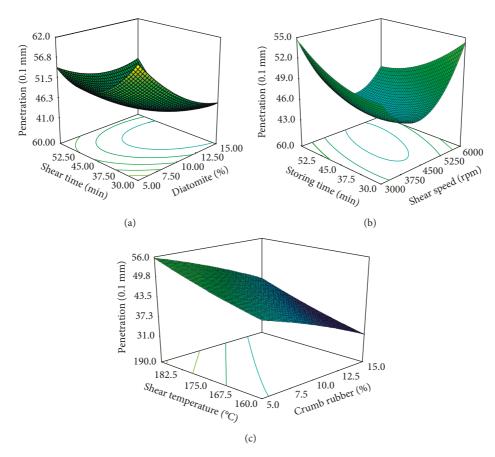


FIGURE 1: Response surface plots for the effect of preparation parameters on penetration. (a) Diatomite content * shear time, (b) shear speed * storing time, and (c) crumb rubber content * shear temperature.

variables on softening point include content of crumb rubber and content of diatomite with p < 0.0001, followed by shear temperature with p < 0.001. Meanwhile, the quadratic term of crumb rubber content has a significant effect with p < 0.01. Furthermore, the interaction terms of (shear speed) * (storing time) have significant effects on softening point with p < 0.01, followed by (crumb rubber content) * (shear speed) with p < 0.05.

The softening points are applied to evaluate the high stability of modified asphalt binder. The higher the softening point is, the better the high susceptibility of asphalt is. The relationships between preparation parameters and softening point were demonstrated in 3D response surface plots, which are shown in Figure 2. As can be seen from the figure, the softening point increases with the increase of diatomite concentration and crumb rubber concentration. However, it decreases with the increase of shear temperature. For the relationships of the softening point with shear time, shear speed, and storing time, they present the trends of increasing firstly and then decreasing.

As discussed above, the polymer chains of rubber particles can absorb the lighter and solvating fractions of binder, and a gel-like structure is generated, which shortens the distances between particles and increases the softening point of binder. Diatomite can effectively absorb the lower molecular group and lower polar aromatic molecular group.

The anchorage structure can improve the high temperature stability of asphalt binder with the increase of diatomite content. As for shear temperature, higher temperature will lead to the breakdown of polymer chains of rubber and crosslink bonds of binder. Therefore, the softening point increases with the increase of shear temperature. Shear speed, shear time, and storing time can strength the gel-like structure and anchorage structure of asphalt binder at the early stage, which make the softening point increase. However, the crosslink bonds between particles are destroyed if the shear time and storing time are longer and the shear speed becomes higher, which decrease the softening point of binder.

3.4. Ductility

3.4.1. Analysis of the Model for Ductility. The ductility model possessed satisfactory levels of R^2 (0.90), Adj. R^2 (0.79), and Adeq. precision (10.875), which were significant at p < 0.0001. R^2 value of the model was higher than 0.8 and Adj. R^2 value was highly close to 0.8, which revealed a close agreement between experimental results and predicted ones. An Adeq. precision of 10.875 indicated that the model can be used to navigate the design space.

As can be seen from the ANOVA results of independent variables for the ductility model, the significant variables

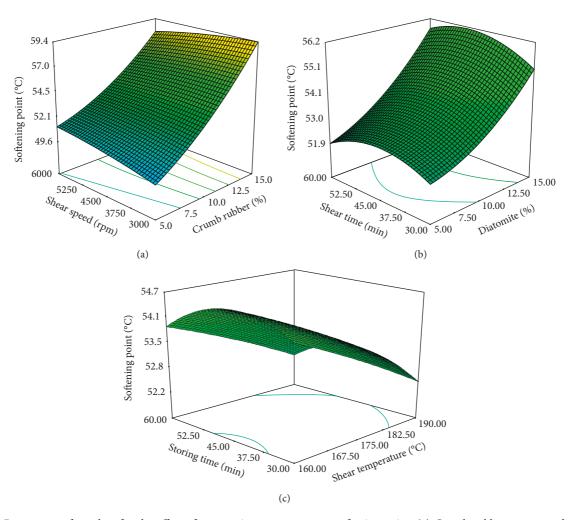


FIGURE 2: Response surface plots for the effect of preparation parameters on softening point. (a) Crumb rubber content * shear speed, (b) diatomite content * shear time, and (c) shear temperature * storing time.

(p<0.05) included X_1 , X_2 , X_5 , X_{14} , X_{25} , X_{46} , X_{33} , X_{44} , and X_{66} . The reduced second-order model in terms of coded factors for ductility was obtained by removing the insignificant variables:

$$\begin{split} Y_3 &= 204.63 - 29.73 \times X_1 - 24.18 \times X_2 + 59.47 \times X_5 \\ &+ 17.71 \times X_1 \times X_4 - 18.84 \times X_{25} - 40.42 \times X_{46} \\ &+ 18.53 \times X_{33} + 42.80 \times X_{44} + 30.59 \times X_{66}. \end{split} \tag{5}$$

3.4.2. Effect of Preparation Parameters on Ductility. As shown in Table 4, the most significant linear variables on ductility include content of crumb rubber and shear temperature with p < 0.0001, followed by content of diatomite with p < 0.001. Meanwhile, the quadratic term of shear speed has a significant effect with p < 0.0001, followed by storing time with p < 0.001 and shear time with p < 0.05. Furthermore, the interaction terms of (shear speed) * (storing time) have significant effects on ductility with p < 0.001, followed by (crumb rubber content) * (shear speed) and (diatomite content) * (shear temperature) with p < 0.01.

Ductility can be used to reflect the low temperature performance of asphalt binder. The relationships between preparation parameters and ductility were demonstrated in 3D response surface plots, which are shown in Figure 3. As can be seen from the figure, the ductility increases with the increase of shear temperature. However, it decreases with the increase of diatomite concentration and crumb rubber concentration. For the relationships of ductility with shear time, shear speed, and storing time, they present the trends of decreasing firstly and then increasing.

The reasons lie in that addition of diatomite and crumb rubber can absorb the lighter fractions of the asphalt and result in large progressive increase of stiffness. The higher the stiffness is, the faster the failure stress reaches [16, 34]. Therefore, the elongation of compound-modified asphalt binder decreases. Another important reason is the stress concentration phenomenon occurring at the interface between the particle and asphalt. Therefore, the reduction of ductility does not indicate that the low temperature performance for crumb rubber and diatomite compound-modified asphalt is unsatisfactory. With the increase of shear temperature, viscosity toughness is reduced, resulting in the increase of

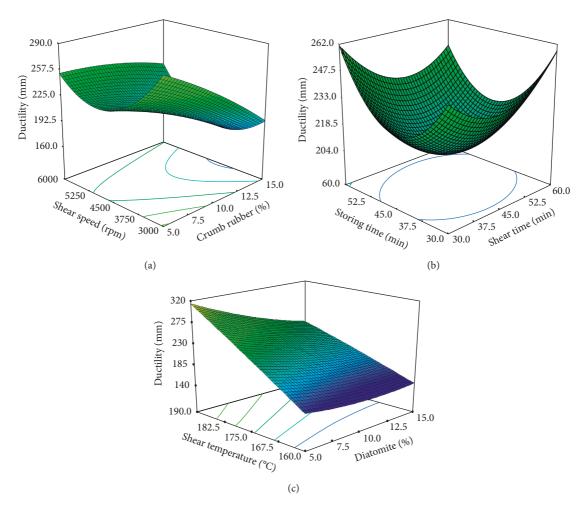


FIGURE 3: Response surface plots for the effect of preparation parameters on ductility. (a) Crumb rubber content * shear speed, (b) storing time * shear time, and (c) shear temperature * diatomite content.

ductility. As for shear speed, shear time, and storing time, they can strength the gel-like structure and anchorage structure of asphalt and increase the stiffness of binder when their values are relatively lower. However, the stable structure will be destroyed when their values reach the critical points. Therefore, ductility decreases firstly and then increases with the increase of shear time, shear speed, and storing time.

3.5. Viscosity

3.5.1. Analysis of the Model for Viscosity. The viscosity model possessed satisfactory levels of R^2 (0.94), Adj. R^2 (0.87), and Adeq. precision (13.392), which were significant at p < 0.0001. R^2 and Adj. R^2 values of the model were higher than 0.8, which revealed a close agreement between experimental results and predicted ones. An Adeq. precision of 13.392 indicated that the model can be used to navigate the design space.

As can be seen from the ANOVA results of independent variables for the viscosity model, the significant variables (p < 0.05) included X_1 , X_3 , X_4 , X_5 , X_{25} , X_{46} , and X_{44} . The reduced second-order model in terms of coded factors for

viscosity was obtained by removing the insignificant variables:

$$\begin{split} Y_4 &= 1907.75 + 1381.14 \times X_1 + 125.24 \times X_3 \\ &- 16.34 \times X_4 - 120.2 \times X_5 - 42.11 \times X_{25} \\ &+ 336.74 \times X_{46} + 100.93 \times X_{44}. \end{split} \tag{6}$$

3.5.2. Effect of Preparation Parameters on Viscosity. As shown in Table 4, the most significant linear variables on viscosity include content of crumb rubber and shear time with $p \le 0.0001$, followed by shear speed with p < 0.001 and shear temperature with p < 0.05. Meanwhile, the quadratic term of shear speed has a significant effect with p < 0.05. Furthermore, the interaction terms of (shear speed) * (storing time) and (diatomite content) * (shear temperature) have significant effects on viscosity with p < 0.01.

The relationships between preparation parameters and viscosity were demonstrated in 3D response surface plots, which are shown in Figure 4. As can be seen from the figure, the viscosities increase with the increase of diatomite concentration and crumb rubber concentration. However, they

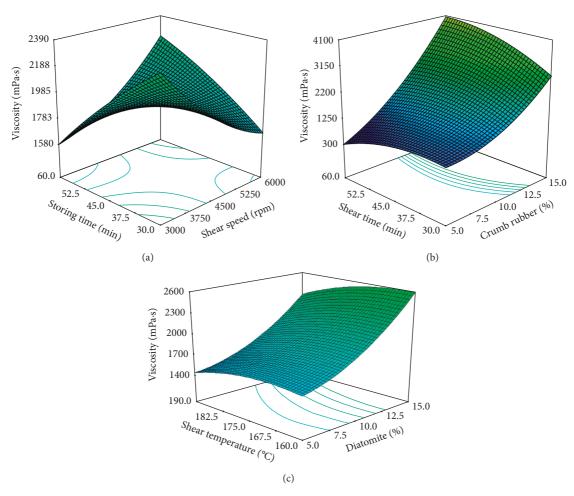


FIGURE 4: Response surface plots for the effect of preparation parameters on viscosity. (a) Storing time * shear speed, (b) crumb rubber content * shear time, and (c) shear temperature * diatomite content.

present a declining trend with the increase of shear time, shear speed, shear temperature, and storing time.

This is because addition of diatomite and crumb rubber into asphalt can form a stable gel-like structure and an anchorage structure. The change in particle size and shape shortens the distances between particles, causing an increase in binder viscosity. If the values of shear temperature, shear speed, shear time, and storing time are too large, rubber particles will be broken down and dissolved into the liquid phase of binder. The crosslink bonds between particles will be destroyed, which will cause a reduction in binder viscosity.

3.6. Elastic Recovery

3.6.1. Analysis of the Model for Elastic Recovery. The elastic recovery model possessed satisfactory levels of R^2 (0.92), Adj. R^2 (0.84), and Adeq. precision (14.374), which were significant at p < 0.0001. R^2 and Adj. R^2 values of the model were higher than 0.8, which revealed a close agreement between experimental results and predicted ones. An Adeq. precision of 14.374 indicated that the model can be used to navigate the design space.

As can be seen from the ANOVA results of independent variables for the elastic recovery model, the significant variables (p < 0.05) included $X_1, X_2, X_{12}, X_{13}, X_{46}, X_{11}$, and X_{33} . The reduced second-order model in terms of coded factors for elastic recovery was obtained by removing the insignificant variables:

$$\begin{split} Y_5 = & \ 43.40 + 13.92 \times X_1 + 0.36 \times X_2 + 2.40 \times X_{12} \\ & + 2.20 \times X_{13} + 5.15 \times X_{46} + 1.16 \times X_{11} - 2.51 \times X_{33}. \end{split} \tag{7}$$

3.6.2. Effect of Preparation Parameters on Elastic Recovery. As shown in Table 4, the most significant linear variables on elastic recovery include content of crumb rubber and content of diatomite with p < 0.0001. Meanwhile, the quadratic term of crumb rubber concentration has a significant effect with p < 0.01, followed by shear speed with p < 0.05. Furthermore, the interaction terms of (crumb rubber concentration) * (shear time) have significant effects on elastic recovery with p < 0.01, followed by (crumb rubber content) * (diatomite content) and (shear speed) * (storing time) with p < 0.05.

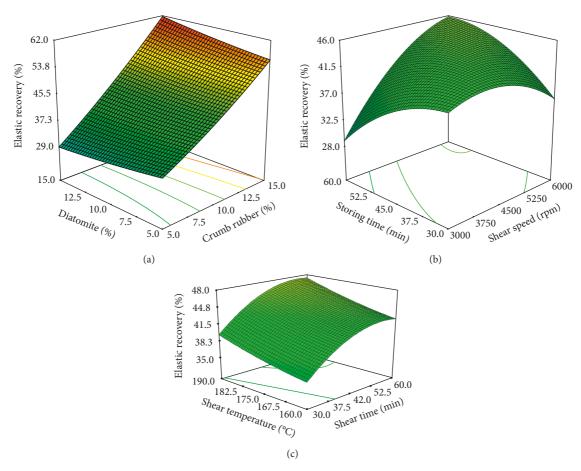


FIGURE 5: Response surface plots for the effect of preparation parameters on elastic recovery. (a) Crumb rubber content * diatomite content, (b) storing time * shear speed, and (c) shear temperature * shear time.

The relationships between preparation parameters and elastic recovery were demonstrated in 3D response surface plots, which are shown in Figure 5. As can be seen from the figure, the elastic recovery increases with the increase of crumb rubber concentration and shear temperature. It increases with the increase of shear time and shear speed but at a decreasing rate. For the relationships of elastic recovery with storing time, they present the trend of increasing firstly and then decreasing slightly. Diatomite concentration can slightly increase the elastic recovery of binder. However, its effect is limited.

As can be seen from the results, the elastic recovery performance of the modified asphalt binder is mainly determined by crumb rubber concentration. The reasons lie in that crumb rubber possesses favorable elastic performance. Additions of crumb rubber into asphalt can strengthen the bonding effect of binder after swelling. The molecular force between particles is increased. Therefore, the ability of elastic recovery is improved. As for diatomite, it is a porous material without good elastic property. Its anchorage structure can enhance the elastic performance of binder to a certain extent. However, the effect is little.

3.7. Penetration Index (PI)

3.7.1. Analysis of the Model for PI. The PI model possessed satisfactory levels of R^2 (0.93), Adj. R^2 (0.85), and Adeq.

precision (14.466), which were significant at p < 0.0001. R^2 and Adj. R^2 values of the model were higher than 0.8, which revealed a close agreement between experimental results and predicted ones. An Adeq. precision of 14.466 indicated that the model can be used to navigate the design space.

As can be seen from the ANOVA results of independent variables for the PI model, the significant variables (p < 0.05) included X_1 , X_2 , X_{14} , X_{23} , X_{46} , X_{22} , X_{33} , and X_{44} . The reduced second-order model in terms of coded factors for PI was obtained by removing the insignificant variables:

$$\begin{split} Y_6 &= -0.59 + 0.62 \times X_1 + 0.16 \times X_2 - 0.13 \times X_{14} \\ &+ 0.19 \times X_{23} + 0.18 \times X_{46} + 0.22 \times X_{22} \\ &+ 0.14 \times X_{33} + 0.22 \times X_{44}. \end{split} \tag{8}$$

3.7.2. Effect of Preparation Parameters on Penetration Index (PI). As shown in Table 4, the most significant linear variables on PI are content of crumb rubber with p < 0.0001, followed by content of diatomite with p < 0.001. Meanwhile, the quadratic term of diatomite concentration and shear speed has a significant effect with p < 0.01, followed by shear time with p < 0.05. Furthermore, the interaction terms of (crumb rubber concentration) * (shear speed), (shear time) * (diatomite content), and (shear speed) * (storing time) have significant effects on PI with p < 0.05.

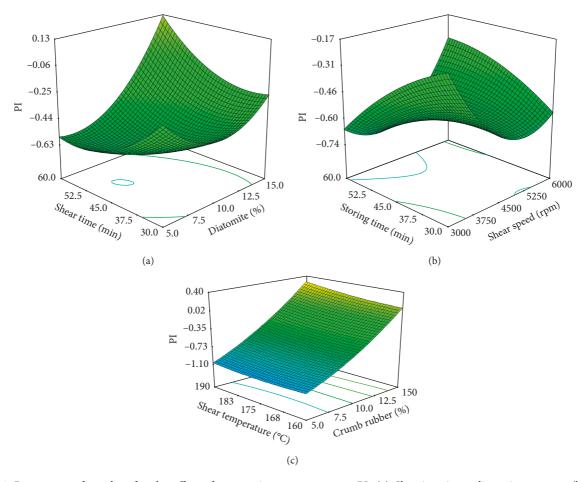


Figure 6: Response surface plots for the effect of preparation parameters on PI. (a) Shearing time * diatomite content, (b) storing time * shear speed, and (c) shear temperature * crumb rubber content.

Table 5: Optimum preparation parameters and corresponding predicted and experimental results.

Factors	X_1	X_2	X_3	X_4	X_5
Optimum value	13.8	6.2	55	5300	183
Responses	\boldsymbol{Y}_1	Y_2	Y_3	Y_4	Y_5
Predicted results	54.15	55.17	307.4	2708	67.63
Experimental results	54.96	54.56	296.6	2814	68.64
Relative error (%)	1.5	1.1	3.5	3.9	1.5

The relationships between preparation parameters and PI were demonstrated in 3D response surface plots, which are shown in Figure 6. As can be seen from the figure, the PI increases with the increase of crumb rubber concentration. It remains relatively stable before 8–10% of diatomite concentration and increases after that. The relationships of PI with shear speed and shear time present the trend of decreasing firstly and then increasing. PI increases with the increase of shear temperature with little amplitude.

The results reveal that addition of crumb rubber and diatomite into asphalt can effectively improve its temperature stability. This is mainly because of the gel-like structure and anchorage structure. As for shear time, shear speed, shear temperature, and swelling time, their influences on

temperature stability of binder are lower than on diatomite concentration and crumb rubber concentration.

3.8. Multiresponse Optimization. Preparation parameters present different effects on properties of crumb rubber and diatomite compound-modified asphalt binder. In order to obtain the modified asphalt binder with satisfactory properties, multiresponse optimization is conducted using the RSM. Taking the modified asphalt used in the cold region, for example, the corresponding optimization criteria are determined according to JTG F40-2004 [35] and "Guide for Design and Construction of Asphalt Rubber and Mixtures" [36] in China. The optimization was carried out by maximizing the softening point, ductility, elastic recovery, and minimizing penetration, while the viscosity is between 1000 and 3000 mPa·s. Viscosity should not be too high because of the workability of modified asphalt binder. PI is not considered because it can be calculated based on penetration and softening point results according to (2). The optimization process is determined and listed in Table 5.

In order to verify the equations as well as the optimum data obtained by the RSM, experimental tests were conducted using the same preparation parameters listed in Table 5. The experimental results are obtained and also shown in Table 5. As shown in this table, the predicted and experimental results possess favorable consistency.

4. Conclusions

In this paper, crumb rubber and diatomite compound—modified asphalt binder was prepared. The effects of preparation parameters on properties of modified asphalt binder were investigated, and multiresponse optimization was conducted using the response surface method. The following conclusions can be obtained:

- (1) Response surface method is a suitable method for preparation parameter analysis and optimization of crumb rubber and diatomite compound-modified asphalt binder, which can effectively reduce the number of groups.
- (2) Crumb rubber concentration presents significant effects on all of the asphalt properties used in this study. Softening points, viscosity, elastic recovery, and PI increase, while penetration and ductility decrease with the increase of crumb rubber concentration. It reveals that the addition of crumb rubber can improve the high temperature susceptibility, viscosity, and elastic recovery ability of binder.
- (3) Diatomite concentration presents significant effects on all of the asphalt properties except elastic recovery. Softening points, viscosity, and PI increase, while penetration and ductility decrease with the increase of diatomite concentration, which presents little influence on elastic recovery of binder.
- (4) Shear temperature presented significant effects on penetration, softening point, viscosity, and ductility. Shear speed, shear time, and storing time have similar effects on binder properties because of their similar mechanism of action.
- (5) Based on the model obtained from the RSM, optimized preparation parameters corresponding to specific criteria can be determined, which possessed favorable accuracy compared with experimental results.

Investigation of other properties such as bending beam rheometer (BBR) and dynamic shear rheometer (DSR) tests of crumb rubber and diatomite compound–modified asphalt binder needs to be conducted in the near future.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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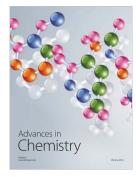


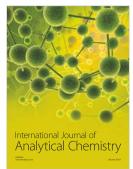














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