1	Innovative use of industrially produced steel slag powders in asphalt mixture to
2	replace mineral fillers
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17	Abstract: Using steel slag to partially replace the natural aggregate in asphalt mixture to produce
18	high-performance asphalt mixture has gained significant interest in recent years as a value-added
19	option to recycle steel slag. However, the poor homogeneity of the properties of steel slag
20	aggregates remains a concern for this recycling approach. In this study, an innovative method of
21	using steel slag powder (SSP) to replace the mineral filler in asphalt mixture was proposed to
22	address this concern. Five fillers, including four SSP fillers, obtained by grinding different steel slag
23	aggregates with an industrialized production line, and one conventional limestone powder (LP)
24	filler, were evaluated. The chemical compositions and micro-morphologies of the SSPs were first
25	characterized to evaluate the material homogeneity and gain insights into the advantages of using
26	SSPs as fillers. Then, asphalt mixtures with different fillers were designed and produced, and their
27	moisture stability, rutting resistance, and low-temperature crack resistance, were characterized. It
28	was found that the industrially produced SSPs possess homogeneous properties, and improved the
29	compatibility between filler particles and asphalt binder, thus enhancing the bonding between
30	asphalt mastic and aggregates. Besides, the asphalt mixtures with SSP fillers showed better

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31 resistance to moisture damage, permanent deformation and low-temperature crack, than the mixture

32 with the LP filler. Therefore, it was concluded that using SSPs as a replacement of mineral fillers in

33 asphalt mixture provides a reliable and value-added solution to recycle steel slag.

34 Keywords: Steel slag powder; material property; filler; asphalt mixture; engineering performance

## 35 **1. Introduction**

36 Asphaltic materials have been widely used as road surfacing materials worldwide (Chang et al., 37 2020; Wang et al., 2020). For example, more than 90% of high-grade pavement in China are paved 38 with asphaltic surfaces. This is mainly because asphalt pavement possesses excellent service 39 performance, such as driving comfort, low noise, good skid resistance and easy maintenance. 40 According to the official statistics of the Ministry of Transport (MOT) of the People's Republic of 41 China, the total mileage of traffic roads has increased about 10.7% during the past five years in 42 China (MOT, 2021), which causes significantly increased consumption of natural resources, such as aggregate and asphalt binder. Meanwhile, China, just like many other countries, is also facing the 43 44 high demand of pavement maintenance. Therefore, developing greener and more sustainable 45 materials for pavement construction and maintenance has become an urgent task. Correspondingly, recycling of appropriate solid wastes in asphalt pavement has become a hot topic, which is a very 46 47 promising way to reduce the exploitation of natural resources. For example, steel slag (Ahmedzade 48 and Sengoz, 2009; Chen et al., 2018; Chen et al., 2015; Chen et al., 2016a; Chen et al., 2016b; Huang et al., 2007; Shen et al., 2009; Xie et al., 2012; Xue et al., 2006), reclaimed asphalt pavement 49 50 (RAP) (Leng et al., 2018b; Xiao et al., 2007; Xiao et al., 2009), waste plastic (Huang et al., 2007) 51 and waste rubber (Huang et al., 2007; Xiao et al., 2009) have been used to partially replace natural 52 aggregate in asphalt mixture; steel slag (Li et al., 2017a; Li et al., 2017b) and flue gas 53 desulfurization ash (Chen et al., 2014a; Chen et al., 2014b) have been applied to replace asphalt 54 filler; and waste plastic (Huang et al., 2007; Leng et al., 2018a; Leng et al., 2018b) and waste rubber 55 (Huang et al., 2007; Xiao et al., 2007) have been recycled as asphalt binder modifiers.

56 Steel slag, generated during steelmaking, is a typical solid waste, which accounts for about 13% 57 of raw steel output (Shen et al., 2009). The total utilization rate of steel slag is less than 30% in 58 China, which is quite low considering the large quantity. Using steel slags as raw materials in 59 asphalt mixture has been proved an effective recycling method (Ahmedzade and Sengoz, 2009; 50 Chen et al., 2018; Chen et al., 2015; Chen et al., 2016a; Chen et al., 2014b; Chen et al., 2014c; 61 Pasetto et al., 2017; Shen et al., 2009; Wu et al., 2007; Xie et al., 2013; Xie et al., 2012; Xue et al., 62 2006). Many previous studies reported that the asphalt mixtures with steel slag aggregate (SSA) 63 presented excellent skid resistance, high-temperature stability and fatigue durability (Ahmedzade 64 and Sengoz, 2009; Chen et al., 2014b; Shen et al., 2009; Wu et al., 2007; Xie et al., 2013; Xue et al., 65 2006). However, different findings have been reported regarding their moisture stability (Chen et al., 66 2020; Chen et al., 2016a; Coomarasamy and Walzak, 1995; Xie et al., 2012). Some laboratory and 67 field research results suggested that SSA was sensitive to the moisture damage (Chen et al., 2020; 68 Chen et al., 2016a; Coomarasamy and Walzak, 1995), especially when the free-thaw effect were 69 considered (Chen et al., 2020; Chen et al., 2016a).

70 The homogeneity of the properties of SSA is relatively poor due to the complex compositions 71 and structures of steel slag, which is one of the possible reasons for the inconsistent research 72 findings on the moisture stability of asphalt mixture with SSA. Coomarasamy and Walzak found 73 that the debonding of the interface between SSA and asphalt mastic in asphalt pavement happened 74 randomly, and the formation of calcium carbonate (CaCO<sub>3</sub>)-rich deposits at the surface of some 75 SSA particles in moist condition caused cracks (Coomarasamy and Walzak, 1995). It was inferred 76 that the compositions of different SSA particles could be different. Some SSA particles have free 77 lime (f-CaO) at the surface, which is the main cause for the CaCO<sub>3</sub>-rich deposits, because f-CaO can 78 be transformed into CaCO<sub>3</sub> by reacting with water and carbon dioxide (CO<sub>2</sub>) in moist condition. It 79 is essentially a carbonation reaction, which causes the expansion problem.

80 In order to eliminate the negative effect of f-CaO on the volume stability of steel slag, the 81 weathering treatment method by placing steel slag in natural environment has been widely used. 82 The original purpose is to make *f*-CaO fully react with water and CO<sub>2</sub> in advance. Chen et al. found 83 that the silicate minerals in steel slag can also participate in the carbonation reaction (Chen et al., 84 2014c). The whole reaction process is illustrated in Fig. 1(a). Hence, for some SSA particles with 85 rich silicate minerals, many CaCO<sub>3</sub> crystal products can be generated. They are adhered to the 86 surface of SSA due to the gelling activity of silicate minerals, which makes the appearance of some 87 SSA particles change from grey-black to grey-white (see Fig. 1(b)). Chen et al. found that these 88 SSA particles with grey-white CaCO<sub>3</sub> product layer is very sensitive to the freeze-thaw damage 89 when used in asphalt mixture. As shown in Fig. 1(c), moisture first damages the bonding interface 90 between asphalt mastic and SSA. Then, moisture infiltrates into the product layer. The frost heave effect of moisture further causes the fracture of the CaCO<sub>3</sub> product layer, which accelerates the
damage of moisture to the bonding interface. Hence, there is risk to use SSA in asphalt mixtures
even after they have gone through weathering treatment.



Fig. 1 (a) Whole carbonation reaction of steel slag during weathering treatment; (b) appearance of
 SSA particles after weathering treatment; (c) freeze-thaw failure process of asphalt mixture with
 SSA (Chen et al., 2020; Chen et al., 2014c)

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98 Considering the potential limitation of utilizing SSA in asphalt mixture, using steel slag powder 99 (SSP) to replace mineral filler in asphalt mixture has been proposed. Although the dosage of filler 100 in asphalt mixture is often less than 10%, it would still be a promising way to reuse steel slag 101 efficiently because of the high demand of asphalt pavement construction and maintenance. So far, 102 very few studies have been conducted on this topic. Li et al. investigated the effects of SSP on the 103 rheological properties of asphalt mastic and the low-temperature performance of asphalt mixture (Li 104 et al., 2017a; Li et al., 2017b). The complex compositions of steel slag, such as metallic iron and 105 some solid solution phases, make steel slag very difficult to be grinded into SSP. Usually, grinding 106 operation will be terminated when obtaining enough powder for experiment purposes. There are 107 still lots of aggregate particles left without being fully grinded. Therefore, SSP prepared in the 108 laboratory by partially grinding some aggregate particles cannot represent the average level of steel 109 slag.

110 To address the concern of the poor homogeneity of the properties of SSA and SSP prepared in

the laboratory, this study aims to develop a new SSP preparation method by fully grinding large volume of SSA with an industrialized production line. The feasibility of innovatively using industrially produced SSP in asphalt mixture to replace mineral filler was fully investigated. The following two research tasks were conducted as shown in Fig. 2:

- (1) The chemical compositions and micro-morphologies of four SSPs were investigated to
  determine the homogeneity of the properties of SSPs and the advantages of using SSPs as
  asphalt fillers.
- (2) Asphalt mixtures with different SSPs were designed by the Superpave method, and their main
   engineering performances, including moisture stability, high-temperature deformation resistance
- 120 and low-temperature crack resistance, were evaluated.



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Fig. 2 Flowchart of the research plan

# 123 2. Materials and methods

# 124 2.1. Raw materials

In this research, five fillers, including four SSPs and one common limestone powder (LP), were used. SSPs were obtained by grinding a large volume of steel slag aggregates (more than 500 kg for each type of steel slag aggregate) with an industrialized production line. Raw steel slag was provided by the China Baowu Steel Group, which is basic oxygen furnace slag. Considering the weathering process during the storage of steel slag, four types of steel slag aggregates were used to produce SSPs, namely, fresh steel slag coarse aggregate (SSCA), weathered SSCA, fresh steel slag fine aggregate (SSFA) and weathered SSFA, and the corresponding SSPs were denoted as SSP<sub>FC</sub>, SSP<sub>WC</sub>, SSP<sub>FF</sub>, SSP<sub>WF</sub>, respectively. The weathering treatment duration was more than 6 months. Limestone powders and limestones for coarse and fine aggregates were both obtained from Yichang, Hubei Province, China. SBS modified asphalt produced by Hubei Guochuang Hi-Tech Material Co., Ltd. was used. The basic properties of the fillers, aggregates and SBS modified asphalt were tested according to the standard test methods (MOT, 2005), and results summarized in Tables 1-3.

37	Table 1 Technical properties of fillers							
	Parameter measured		SSP <sub>FC</sub>	SSP <sub>wc</sub>	SSP <sub>FF</sub>	SSP <sub>WF</sub>	LP	Requirements according to Chinese specification (MOT, 2004)
	Apparent der	sity (g/cm <sup>3</sup> )	3.468	3.477	3.322	3.326	2.707	≥ 2.5
	Percent passing (%)	0.6 mm	100	100	100	100	100	100
		0.15 mm	92.8	92.5	92.1	92.3	92.7	90-100
		0.075 mm	85.9	84.9	86.9	85.4	86.8	75-100

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#### **Table 2** Technical properties of aggregates

	Coarse ag	ggregate	Fine aggregate	Requirements according to Chinese specification (MOT, 2004)
Parameter measured	16-9.5 m	m 9.5-4.75 mm	4.75-0 mm	
Apparent specific gravity	2.694	2.688	2.698	≥ 2.5
Water absorption (%)	0.5	0.4		$\leq$ 2.0
Crush value (%)	20.7			$\leq 28$
Los Angeles abrasion (%)	21.5	21.5		≤ 30
Flakiness and elongation (%)	6.6	7.3		≤18
Fine aggregate angularity (s)			52	$\geq$ 30
Sand equivalent (%)			65	$\geq 60$

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## Table 3 Technical properties of SBS modified asphalt

Parameters	SBS modified asphalt	Requirements according to Chinese specification (MOT, 2004)	
Penetration (25°C; 0.1mm)	63	60-80	
Softening point (°C)	76	≥ 55	
Ductility (5°C; cm)	45	$\geq$ 30	
Elasticity resume (25°C; %)	71	$\geq$ 65	

#### 140 2.2. Experimental methods

#### 141 2.2.1. Comparative analysis of the properties of SSPs

The chemical compositions and micro-morphologies of the SSPs were first characterized. The chemical compositions were analyzed by ethylene glycol-EDTA chemical titration and x-ray fluorescence (XRF). The former was to determine the content of *f*-CaO, and the later was for determining the average chemical compositions. Although *f*-CaO in steel slag is finally transformed into CaCO<sub>3</sub> after weathering treatment, some intermediate product, Ca(OH)<sub>2</sub>, still exist. Ca(OH)<sub>2</sub> can also participate in the chemical titration reaction, as shown in the following equations:

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$$CaO + C_2H_6O_2 \rightarrow (CH_2O)_2Ca + H_2O$$
(1)

149 
$$Ca(OH)_2 + C_2H_6O_2 \rightarrow (CH_2O)_2Ca + 2H_2O$$
(2)

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$$C_{10}H_{14}N_2Na_2O_8 + (CH_2O)_2Ca \rightarrow C_{10}H_{14}N_2CaO_8 + HOCH_2CH_2OH$$
 (3)

Therefore, chemical titration gives the total content of  $Ca^{2+}$  attributing to *f*-CaO and Ca(OH)<sub>2</sub>. In this research, an auxiliary thermal gravimetric (TG) analysis device was used to determine the content of Ca(OH)<sub>2</sub> based on the mass change of each SSP in the decomposition temperature range. Finally, the content of *f*-CaO in SSP can be determined. The micro-morphologies of SSPs were observed by scanning electron micrograph (SEM).

The homogeneity of the properties of SSPs were analyzed based on the characterization results. SSA and LP were used as the control groups. In each characterization test, five replicates were prepared and tested. For each filler, the five powder samples were randomly picked out from the industrially produced SSPs and LP. For fresh SSCA and weathered SSCA, five particles with size of 19 mm were also randomly obtained from a large volume of SSA. For fresh SSFA and weathered SSFA, five samples with each composed of ten randomly selected particles with a size of 2.36 mm were used. In chemical titration and XRF analysis, they were grinded into powders in advance.

163 2.2.2. Engineering performance evaluation of asphalt mixtures

In this study, the Superpave design method was applied to design the mixtures, and the standard test methods in accordance with the Chinese technical specifications were adopted to evaluate the engineering performances of the asphalt mixtures (MOT, 2011). High-temperature deformation resistance, low-temperature crack resistance and moisture damage resistance were determined by the wheel tracking test, three-point bending test, and retained Marshall stability (RMS) test and tensile strength ratio (TSR) test, respectively. In addition, the volume stability test of asphaltmixture was also included as a supplementary investigation.

171 2.2.2.1. Design of asphalt mixture by the Superpave procedure

172 Considering that only the filler types were different among the designed asphalt mixtures, each 173 asphalt mixture was marked by the filler type, namely SSPFC mixture, SSPwC mixture, SSPFF 174 mixture, SSP<sub>WF</sub> mixture and LP mixture. The proportions of the coarse aggregate, fine aggregate 175 and filler in the mineral mixture were 46%, 50% and 4%, respectively. Although the particle size 176 distributions of different fillers were not exactly same (see Table 1), they showed insignificant 177 effect on the gradations of the mineral mixtures due to their low dosages. As a result, the gradations 178 of these five mineral mixtures were almost the same. The used gradation is shown in Fig. 3. The 179 optimum asphalt contents (OACs) of the asphalt mixtures were determined to be 4.9 %. It indicated 180 that filler type has little effect on the OACs of the asphalt mixtures.



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# 183 2.2.2.2. Moisture damage resistance evaluation

Moisture stabilities of asphalt mixture are often determined by the RMS test and TSR test, which are conducted under hot water immersing damage condition and freeze-thaw cycle damage condition, respectively. The most noteworthy issue of reusing steel slag in asphalt mixture is the volume stability in hot and moist condition. Therefore, the volume stabilities of SSP asphalt mixtures under hot water immersing condition were also investigated in this research.

*Hot water immersing damage*. Specimens with a diameter of 100 mm and thickness of 63.5 mm
and 4% air void content were used. The volume stability of the SSP asphalt mixture was checked by

immersing specimens in a 60 °C water bath and recording the volumes of the specimens every 24 h.
The immersion operation lasted for 72 h. The volume expansion percentage of each specimen was
calculated by the following equation:

$$p_{ve} = \frac{v_i - v_0}{v_0} \times 100\%$$
(4)

where  $p_{ve}$  is the volume expansion percentage of specimen, %;  $v_0$  is the original volume of the specimen, cm<sup>3</sup>; and  $v_i$  is the volume of the specimen after being immersed in hot water for *i* h, cm<sup>3</sup>.

For the RMS test, the same specimens for the volume stability tests were used, which were divided into four groups. One was the control group, and the other three were conditioned groups. Specimens in the conditioned groups were immersed in a water bath of 60 °C for 24 h, 48 h and 72 h, respectively. The Marshall stabilities of the conditioned groups and control group were measured to determine the RMS using the following equation:

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$$RMS = \frac{MS_n}{MS_0} \times 100\%$$
<sup>(5)</sup>

where  $MS_0$  is the average Marshall stability of the control group, kN; and  $MS_n$  is the average Marshall stability of the conditioned group after being immersed in hot water for *n* h, kN.

*Freeze-thaw cycle damage.* Cylindrical specimens with a diameter of 100 mm and thickness of 63.5 mm and air void content of 6% were prepared for the freeze-thaw cycle tests. The three conditioned groups were subjected to freeze-thaw damage for 1 cycle, 2 cycles and 3 cycles, respectively. One freeze-thaw cycle refers to freeze the specimens in a freezer at -18 °C for 16 h , and then thaw them in the water bath at 60 °C for 24 h. The indirect tensile strength (ITS) of each specimen was tested, and the following equation was used to measure the TSR:

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$$TSR = \frac{ITS_{h}}{ITS_{0}} \times 100\%$$
(6)

where  $ITS_0$  is the average ITS of the control group, MPa; and  $ITS_h$  is the average ITS of the conditioned group after freeze-thaw damage for *h* cycles, MPa.

### 214 2.2.2.3. *High-temperature deformation resistance*

Asphalt concrete slabs with 300 mm×300 mm×50 mm in size were used to evaluate the hightemperature performance of asphalt mixture. A simple wheel tracking device with a wheel load of 0.7 MPa was used. The wheel (50 mm width) moves back and forth along the center area of the specimens with a speed of 42 passes/min. Three test temperatures (50 °C, 60 °C and 70°C) were considered. The high-temperature stability index of the asphalt mixture, named dynamic stability,was determined by the following equation:

 $DS = \frac{15 \times s}{d_{60} - d_{45}} \tag{7}$ 

where *DS* is the dynamic stability, pass/mm; *s* is the wheel speed, pass/min;  $d_{45}$  and  $d_{60}$  are rutting depths of specimens corresponding to 45 min and 60 min, respectively, mm.

#### 224 2.2.2.4. Low-temperature crack resistance

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Low-temperature crack resistance of asphalt mixture was evaluated by the three-point bending test. The dimension of the beam specimens was 250 mm×30 mm×35 mm. UTM-30 with automatic temperature control and data acquisition system was utilized to bend the beam specimens at a vertical loading rate of 50 mm/min. The test temperature was -10 °C. The flexural strength and strain were calculated by the following equations:

$$\sigma(t) = \frac{3lf(t)}{2bh^2} \times 10^3 \tag{8}$$

$$\varepsilon(t) = \frac{6hd(t)}{l^2} \times 10^6 \tag{9}$$

where  $\sigma(t)$  is the flexural strength of beam specimen, MPa; f(t) is the value of force loaded on beam specimen, kN; *l*, *b* and *h* are the spanning length, width and height of beam specimen, respectively, mm;  $\varepsilon(t)$  is the strain of beam specimen,  $\mu\varepsilon$ ; and d(t) is the vertical deflection of beam specimen, mm.  $\sigma(t)$ , f(t),  $\varepsilon(t)$ , and d(t) are the functions of testing time *t*.

The Chinese technical specification for highway asphalt pavements construction focuses on the strain value when determining the low-temperature performance of asphalt mixture (MOT, 2004). But flexural strength may also affect the low-temperature performance of asphalt pavement. For example, higher flexural strength gives asphalt pavement better resistance to traffic load damage. Therefore, besides strain value, fracture energy, which involves both flexural strength and strain, was also calculated in this study using the following equation:

 $fe = 10^{-3} \int_{0}^{\varepsilon_{f}} \sigma d\varepsilon$  (10)

243 where *fe* is fracture energy, kJ/m<sup>3</sup>; and  $\varepsilon_f$  is the failure strain when  $\sigma$  reaches the maximum value,  $\mu\varepsilon$ .

#### **3. Results and discussions**

- 245 3.1. Properties of SSPs
- 246 3.1.1. Homogeneity analysis

247 The contents of f-CaO in SSA particles and SSPs determined by the chemical titration method are 248 shown in Fig. 4. It can be seen that, for aggregate particles, either SSCA or SSFA, the contents of f-249 CaO presented significant fluctuations. The error bars showed a standard deviation of 1.4%, 2.8%, 250 1.6% and 1.3% for randomly selected fresh SSCA, weathered SSCA, fresh SSFA and weathered 251 SSFA particles, respectively. The average contents of f-CaO in the tested particles also displayed a 252 strange trend: compared with the fresh SSCA and SSFA particles, weathered aggregate particles 253 showed higher average contents of f-CaO, which contradicts to the expectation. Theoretically, the 254 contents of f-CaO in weathered SSA particles should be lower. The compositions of steel slag are 255 very complex, and the nonuniform distribution behaviors of some compositions can further explain 256 the strange results shown in Fig. 4.



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259 The cross-sectional image of steel slag block indicated that some compositions of steel slag, such as metallic iron and *f*-CaO, were clustered rather than uniformly distributed (see picture a in Fig 5). 260 261 The binary image shown in Fig. 5(b) more clearly illustrates their clustered distributions. Hence, 262 zones of *f*-CaO enrichment would be included in some aggregate particles when preparing SSA by 263 crushing steel slag blocks, and the contents of *f*-CaO in some aggregate particles would vary 264 significantly, which lead to large-sized error bars of f-CaO content. Especially for the weathered 265 SSCA, several zones of f-CaO enrichment have been observed when grinding two of the selected 266 five particles.



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**Fig. 5** (a) Cross-sectional appearance of steel slag block; (b) binarization processed cross-sectional image; (c) localized expansion phenomenon of weathered SSCA based asphalt mixture

270 Although weathering treatment is a convenient way to reduce the expansion behavior caused by 271 f-CaO, the carbonation reaction only occurs at the surface of SSA particles. So, the contribution of 272 weathering treatment on the reduction of total f-CaO content is not significant. The content of f-CaO 273 is highly dependent on the possible f-CaO enrichment region in the internal of SSA particles. Hence, 274 the average contents of f-CaO in selected weathered SSA particles were not necessarily lower than 275 those of selected fresh SSA particles considering the random distribution of f-CaO enrichment 276 regions in different particles. As Fig. 4 shows, weathered SSCA and SSFA particles even possessed 277 higher contents of f-CaO than fresh ones. The localized expansion cracks of weathered SSCA based 278 asphalt mixture also supported this finding. Fig. 5(c) shows the broadside image of a cored asphalt 279 mixture sample with weathered SSCA after hot water immersing damage. Localized expansion of 280 weathered steel slag particle caused by f-CaO can be observed, indicating that the enrichment 281 regions of *f*-CaO in the internal of steel slag particles remained even after weathering treatment.

282 The situation was quite different when SSPs were used. The four SSPs displayed very stable 283 average contents of f-CaO. The error bars showed a standard deviation of 0.3%, 0.2%, 0.2% and 0.3% 284 for SSP<sub>FC</sub>, SSP<sub>WC</sub>, SSP<sub>FF</sub> and SSP<sub>WF</sub>, respectively. It indicated that grinding aggregate particles into 285 SSPs was an effective way to overcome the significant variations of the contents of f-CaO in 286 different SSA particles. Therefore, chemical titration results obtained based on SSPs were reliable 287 to show the actual average level of f-CaO in steel slag. Particularly, there was no significant 288 difference between the contents of f-CaO in SSP<sub>FC</sub> and SSP<sub>WC</sub>, which also proved that carbonation 289 reaction occurring at the surface of SSCA had minor effects on the average f-CaO content in steel 290 slag. However, the contents of f-CaO for the SSPs prepared by grinding fresh SSFA and weathered 291 SSFA were very different. As Fig. 4 shows, the f-CaO content of SSP<sub>WF</sub> was 18% lower than that of

292 SSP<sub>FF</sub>. This is because the carbonation reaction efficiency of SSFA is higher than that of SSCA, 293 contributed by the following two reasons: 1) the zones of *f*-CaO enrichment are more easily 294 exposed to the surface of SSFA; and 2) the specific surface area of SSFA is larger.

The average chemical compositions of SSA particles and SSPs were determined by XRF. Some main chemical compositions are shown in Fig. 6. From Fig. 6a, it can be seen that, even for the same type of SSA, different particles presented significant difference in the contents of CaO, SiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub>, because of the complex compositions of aggregate particles. Fig. 6b shows that unlike the XRF results of SSA particles, the variations in the contents of the main chemical compositions of each SSP are very small as evidenced by the short error bars. Hence, XRF tests on SSPs are able to provide more reliable average chemical compositions of steel slag.



Fig. 6 Main chemical compositions: (a) SSAs; (b) SSPs

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304 From Fig 6(b), it can also be observed that the CaO contents of all SSPs were above 40%, while 305 the corresponding f-CaO contents were less than 10% (see Fig. 4), which means that a large amount 306 of CaO existed in other forms. Two forms of CaO have been reported. One was to form silicate 307 minerals (dicalcium silicate and tricalcium silicate) with  $SiO_2$ , accordingly, the contents of  $SiO_2$ 308 were also substantial as shown in Fig. 6(b). For SSP<sub>FC</sub> and SSP<sub>WC</sub>, the differences of CaO content 309 and SiO<sub>2</sub> content were 1.7% and 0.7%, respectively. And which were 5.2% and 2.1%, respectively, 310 for SSP<sub>FF</sub> and SSP<sub>WF</sub>. It also supported the conclusion that the carbonation reaction of SSFA during 311 the weathering process ran more thoroughly. More silicate minerals and f-CaO in SSFA participated 312 in the carbonation reaction, and the relative contents of CaO and SiO<sub>2</sub> decreased because of 313 absorbing  $CO_2$  in the air. The other was to form a complex solid solution with some other oxides. 314 The most important oxides in solid solution were these related to Fe element such as FeO and Fe<sub>2</sub>O<sub>3</sub>. 315 Besides ferrous and iron oxides, a great amount of Fe element existed in steel slag as metallic iron, 316 as shown in Fig 5(a) and (b). They can be identified quickly because of some twills with the 317 metallic luster. Fig 5(a) and (b) also showed that the metallic irons were clustered in steel slag 318 blocks. Actually, many zones of metallic iron enrichment cannot be well-sorted out when crushing 319 steel slag blocks although magnetic separation devices are configured. This is because some 320 metallic iron is wrapped or non-magnetic. As a result, some SSA particles inevitably contain 321 clustered metallic iron. And it is not easy to crush steel slag blocks containing metallic iron, 322 therefore, coarse aggregate particles are more likely to occupy clustered metallic iron. It can explain 323 why SSP<sub>FC</sub> and SSP<sub>WC</sub> presented higher densities than SSP<sub>FF</sub> and SSP<sub>WF</sub> (see Table 1). In fact, the 324 contents of oxides given by XRF analysis are computed based on the concentrations of different 325 elements. So the contents of Fe<sub>2</sub>O<sub>3</sub> shown in Fig. 6 did not represent the actual amount of Fe<sub>2</sub>O<sub>3</sub> in steel slag. To some extent, they showed the total contents of Fe element related compositions such 326 as metallic iron and oxides. So, as shown in Fig 6(b), compared to SSP<sub>FF</sub> and SSP<sub>WF</sub>, the contents of 327 328 Fe<sub>2</sub>O<sub>3</sub> in SSP<sub>FC</sub> and SSP<sub>WC</sub> given by XRF analysis were higher due to the contribution of more 329 likely clustered metallic iron in coarse aggregate particles. As a result, the contents of other 330 chemical compositions got lower relatively, such as lower content of CaO in SSP<sub>FC</sub> than that in SSPFF. 331

332 The SEM test results indicated that the selected SSCA particles and SSFA particles all showed 333 various micro-morphologies, and the main texture characteristics were almost the same. Therefore, 334 only the micro-morphologies of SSCA particles before and after weathering treatment were 335 analyzed in this research. As shown in Fig. 7(a), fresh SSCA particles mainly possessed three 336 different surface structures, namely, dense structure, honeycomb structure and lamella structure. 337 Some pores can be observed in the dense structure zone. The weathering process significantly 338 modified the micro-morphologies of SSCA particles. It can be seen that, after weathering treatment, 339 the dense structure zone was covered by carbonation reaction products of CaCO<sub>3</sub> (see Fig. 7(b)), 340 indicating that the dense structure zone was rich in active minerals, which mainly included f-CaO 341 and silicate minerals as shown in Fig. 1(a). And some small-sized products were embedded in the 342 honeycomb structure zone (see Fig. 7 (c)). It suggested that the honeycomb structure zone was lack 343 of active minerals. Many bar-shaped gypsum crystals were presented in the lamella structure zone 344 (see Fig. 7(d)). They were quite different from CaCO<sub>3</sub> products. Therefore, except for carbonation

345 reaction, some other reactions also occurred during weathering process although it was not the main 346 reaction type. The various micro-morphologies of fresh and weathered SSCA particles also support 347 the conclusion that the compositions and structures of SSA particles are complex and changeable.



Fig. 7 The micro-morphologies of SSCA and SSPs: (a) Fresh SSCA; (b) dense zone in Fresh SSCA
after weathering treatment; (c) honeycomb structure zone in Fresh SSCA after weathering treatment;
(d) lamella structure zone in fresh SSCA after weathering treatment; (e) SSP<sub>FC</sub>; (f) SSP<sub>WC</sub>; (g)
SSP<sub>FF</sub>; (h) SSP<sub>WF</sub>; (i) LP

The homogeneity of the micro-morphologies of SSPs was much better than that of SSA particles. Fig. 7(e) to Fig. 7(h) displayed that the micro-morphologies of small-sized particles in four different SSPs were extremely similar. Their shapes were irregular, and the surface textures were very coarse. As a result, a conclusion similar to that in chemical composition analysis can be obtained. Compared to the changeable micro-morphologies of aggregate particles, grinding them into SSPs was also an effective way to homogenize the micro-morphologies of steel slag.

359 3.1.2. Advantages of using SSPs as asphalt fillers

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360 The bonding behavior between aggregate and asphalt mastic directly influences the performances

361 of asphalt mixture, especially the moisture damage resistance. Alkaline fillers such as Portland 362 cement, hydrated lime was normally used to improve the engineering performances when 363 constructing asphalt pavements in China. As a result, alkaline fillers are more than welcome. Mason 364 has proposed an equation,  $M=w(CaO)/[w(SiO_2)+w(P_2O_5)]$ , to compute the alkalinity of steel slag 365 (Mason, 1994). Steel slag is normally classified into three groups according to the value of 366 alkalinity. They are low alkalinity slag (M<1.8), intermediate alkalinity slag (1.8<M<2.5) and high 367 alkalinity slag (M>2.5) (Wang et al., 2011). The alkalinities of four SSPs in this research were 368 shown in Fig.8, and they were determined based on the average chemical compositions of SSPs 369 given by XRF. It can be seen that all alkalinities of the four SSPs were above 2.5, and it suggested 370 that they were high alkalinity steel slags. It is well known that the common LP filler mainly 371 contains CaCO<sub>3</sub> minerals. The carbonation of *f*-CaO and silicate minerals in SSPs also resulted in 372 the generation of CaCO<sub>3</sub> minerals. It suggested that SSPs were more alkaline. Alkaline SSPs 373 presented advantages when used as asphalt fillers. They improved the compatibility between filler 374 particles and weak acid asphalt binder.

Specifically in Fig 8, although the carbonation reaction of some *f*-CaO and silicate minerals has absorbed some CO<sub>2</sub> during weathering treatment, it affected the alkalinity values of SSPs little. The differences of alkalinity values between SSP<sub>FC</sub> and SSP<sub>WC</sub>, SSP<sub>FF</sub> and SSP<sub>WF</sub> were small. Actually, the content of P<sub>2</sub>O<sub>5</sub> in steel slag is less than 1%, the alkalinity is mainly determined by the ratio of w(CaO) to  $w(SiO_2)$ . Fig. 6(b) showed that the contents of CaO and SiO<sub>2</sub> in SSCA and SSFA both decreased after weathering treatment, especially that in SSFA. Therefore, low contents of *f*-CaO in SSP<sub>WC</sub> and SSP<sub>WF</sub> does not necessarily lead to low alkalinity values.





384 While, Fig. 8 also showed that the alkalinity values of SSP<sub>FF</sub> and SSP<sub>WF</sub> were about 10% lower 385 than that of  $SSP_{FC}$  and  $SSP_{WC}$ . This may be due to the contribution of more silicate minerals in 386 SSFA. As stated in the section of 3.1.1, compared to SSCA, SSFA was more likely to possess small 387 amounts of clustered metallic iron. As a result, the contents of other minerals will be relatively 388 higher, such as silicate minerals. Silicate minerals in steel slag mainly include dicalcium 389 silicate( $C_2S$ ) and tricalcium silicate ( $C_3S$ ), and the ratios of w(CaO) to  $w(SiO_2)$  are 1.87 and 2.8, 390 respectively. The ratios of w(CaO) to  $w(SiO_2)$  in SSP<sub>FC</sub> and SSP<sub>WC</sub> were around 3.0. Therefore, 391 even the increased silicate minerals in SSFA were all C<sub>3</sub>S, the ratios of w(CaO) to w(SiO<sub>2</sub>) in SSP<sub>FF</sub> 392 and SSP<sub>WF</sub> would be still lower than that in SSP<sub>FC</sub> and SSP<sub>WC</sub>.

The micro-morphologies of SSPs also gave an advantage in terms of using them as asphalt fillers. Pictures e to i in Fig.7 presented that the small particles in SSPs possessed rich convex and corrugated structures. While, the micro-surfaces of small particles in common LP were much smoother. The rich convex and corrugated micro-surfaces increased the specific surface area of SSPs and also raised the compatibility between SSPs and asphalt binder, which was beneficial for the stable bonding interaction between SSPs asphalt mastic and aggregate.

399 *3.2. Performances of asphalt mixtures* 

# 400 *3.2.1. Moisture damage resistance*

401 As Fig. 9 shows, the average volume expansion percentage of all asphalt mixtures were no more 402 than 0.6% even after hot water damage for 72 h. The volume expansion percentage of asphalt 403 mixture containing steel slag should be less than 1.5% according to the Chinese specification (MOT, 2004). Therefore, using SSPs as asphalt filler can give asphalt mixture satisfactory volume stability. 404 405 Even for the LP asphalt mixture, the volume expansion percent ranged from 0.45% to 0.56% with 406 the increase of hot water damage time. Although the volume expansion percentage of asphalt 407 mixtures containing different SSP showed some fluctuations when subjected to same amount of hot 408 water immersion time, they were not obviously different from that of the LP asphalt mixture. The 409 change interval for the volume expansion percent of SSPs asphalt mixtures was 0.39% to 0.6% 410 when hot water damage time increased from 24 h to 72 h. Unlike the weathered SSCA asphalt 411 mixture that presented a localized expansion phenomenon after hot water damage (see Fig. 5c), no 412 cracks can be observed around the area of SSPs asphalt mastics. It indicated that the volume 413 expansion of SSPs asphalt mixtures was not caused by the carbonization of f-CaO in SSPs. Instead,







Fig. 9 Volume stability test results of asphalt mixtures

417 The RMS results of asphalt mixtures are shown in Fig. 10, which indicated that the Marshall 418 stabilities of asphalt mixtures were sensitive to the hot water damage. There was a significant drop 419 in RMS value of each asphalt mixture when increasing the hot water immersing time. The RMS 420 values of SSPFC mixture, SSPWC mixture, SSPFF mixture, SSPWF mixture and LP mixture decreased 421 11.9%, 11.4%, 10.1%, 10.6% and 12.6%, respectively, when hot water damage time reached 72 h. 422 It indicated that SSPs played positive roles in improving the hot water damage resistance of asphalt 423 mixtures. Fig. 10 also displayed that the weathering treatment has insignificant effects on the hot 424 water damage resistance of asphalt mixtures. The differences of RMS values between SSPFC 425 mixture and SSP<sub>WC</sub> mixture, SSP<sub>FF</sub> mixture and SSP<sub>WF</sub> mixture after hot water damage for the same 426 time were very small. While, from the perspective of slag aggregate types, these two categories of 427 SSPs prepared by grinding SSCA (SSP<sub>FC</sub> and SSP<sub>WC</sub>) and SSFA (SSP<sub>FF</sub> and SSP<sub>WF</sub>) respectively 428 performed differently in maintaining the Marshall stabilities of asphalt mixtures. Although the RMS 429 values of these four asphalt mixtures containing different SSP were very close after short-term hot 430 water damage (24 h), the difference of the RMS values was gradually magnified with the increase 431 of hot water damage time. Compared to SSPFC and SSPWC, the asphalt mixtures containing SSPFF 432 and SSP<sub>WF</sub>, showed higher RMS values after longtime hot water damage. It suggested that SSP<sub>FF</sub> 433 and SSP<sub>WF</sub> improved the durability of asphalt mixture in a high-temperature and moist environment. 434 Therefore, the natural difference of material properties in SSCA and SSFA affected the hot water 435 damage resistance of SSPs asphalt mixtures more obviously.





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Fig. 11 presents the TSR results of SSPs asphalt mixtures, which showed similar trends as those of RMS. It can be seen that the indirect tensile strength of each asphalt mixture was also very sensitive to the freeze-thaw cycle damage. The TSR values of SSP<sub>FC</sub> mixture, SSP<sub>wc</sub> mixture, SSP<sub>FF</sub> mixture, SSP<sub>wF</sub> mixture and LP mixture decreased 19.4%, 19.1%, 17.6%, 17.1% and 19.7%, respectively, after suffering freeze-thaw damage for 3 cycles. In general, SSPs asphalt mixtures presented comparable or better freeze-thaw cycle damage resistance than LP asphalt mixture. Compared to LP, the advantages given by SSP<sub>FC</sub> and SSP<sub>wc</sub> were quite limited, while those 447 provided by SSP<sub>FF</sub> and SSP<sub>WF</sub> were significant. Similar to the RMS results, TSR values of SSP<sub>FC</sub> 448 mixture and SSP<sub>WC</sub> mixture, and of SSP<sub>FF</sub> mixture and SSP<sub>WF</sub> mixture after freeze-thaw 449 conditioning were very close. Hence, the TSR results also supported that the natural difference of 450 material properties in SSCA and SSFA influenced the freeze-thaw cycle damage resistance of SSPs 451 asphalt mixtures more.

452 Although different types of SSP showed different abilities in maintaining the RMS and TSR 453 values of asphalt mixtures, all SSPs always did better than LP, as shown in Fig. 10 and Fig. 11. It is 454 widely agreed that the cohesion failure of the interior of asphalt mastic and the adhesion failure of 455 the asphalt mastic and aggregate are the main damage forms of water to the asphalt mixture. For 456 asphalt mastic, when using SSPs to replace common LP filler, the high alkalinity, rich convex and 457 corrugated morphologies of SSPs improved the compatibility between filler particles and weak acid 458 asphalt. On the one hand, the asphalt mastic itself was strengthened, and on the other hand, the 459 adhesion performance of asphalt mastic and aggregate was also enhanced. As a result, SSPs played 460 very positive roles in improving the moisture damage resistance of asphalt mixture.

## 461 *3.2.2. High-temperature deformation resistance*

462 The high-temperature deformation resistance of the Superpave asphalt mixture was evaluated by 463 the wheel tracking test. The dynamic stabilities of asphalt mixtures computed based on wheel 464 tracking test results are presented in Fig. 12. It is clear that SSPs strengthened the deformation resistance of asphalt mixtures at different test temperatures. At 50 °C, the dynamic stabilities of 465 466 asphalt mixtures containing four different SSP were very close, which were approximately 10% 467 higher that of LP asphalt mixture. But with the increase of test temperature, the contributions of 468 four SSPs became different. SSP<sub>FF</sub> and SSP<sub>WF</sub> performed better in maintaining the deformation 469 resistance of asphalt mixtures at 60 °C and 70 °C. It indicates that the SSPs manufactured by 470 grinding SSFA played a positive role in improving the deformation resistance of asphalt mixture at 471 higher temperatures, which may also be contributed by the natural difference of material properties 472 in SSCA and SSFA.



# 473



## Fig. 12 Dynamic stability results of asphalt mixtures

# 475 *3.2.3. Low-temperature crack resistance*

The low-temperature crack resistance test results, i.e., low-temperature strain and fracture energy, 476 of each asphalt mixture are presented in Fig. 13. It can be seen that the effects of SSPs on the low-477 478 temperature strain and fracture energy were inconsistent. The strain values of SSP<sub>FC</sub> mixture, 479 SSP<sub>WC</sub> mixture, SSP<sub>FF</sub> mixture and SSP<sub>WF</sub> mixture were 6.2%, 8.8%, 4.9% and 2.9% lower than 480 that of LP mixture, respectively, indicating that SSPs have hardened the asphalt mixture at low 481 temperatures. But SSP<sub>FF</sub> and SSP<sub>WF</sub> performed better in maintaining the low-temperature strain than 482 SSP<sub>FC</sub> and SSP<sub>WC</sub>. The effect of SSPs on the fracture energy of asphalt mixtures was just the 483 opposite. Fracture energy is determined based on both flexural strength and strain of asphalt 484 mixture, as well as their correlation, so it is a more scientific index to determine the low-485 temperature performance of asphalt mixture. Fracture energy index has also been widely adopted in previous research (Wang et al., 2021; Zhang et al., 2019). Hence, the low-temperature bending test 486 487 results indicated that SSPs improved the low-temperature crack resistance of asphalt mixtures in 488 terms of fracture energy index, even though they have slightly lowered the strain values of asphalt 489 mixtures.



491

Fig. 13 Low-temperature bending test results of asphalt mixtures

## 492 **4. Conclusions**

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In this study, an innovative method of using SSP to replace mineral filler in asphalt mixture was proposed to address the concern of poor property homogeneity of steel slag aggregates. Four SSPs, including SSP<sub>FC</sub>, SSP<sub>WC</sub>, SSP<sub>FF</sub> and SSP<sub>WF</sub>, were prepared by grinding large volume of fresh SSCA, weathered SSCA, fresh SSFA and weathered SSFA, respectively, using an industry production line, and investigated through a series of laboratory tests. Based on the results of the laboratory tests, the following major findings have been obtained:

- (1) Unlike the nonhomogeneous chemical compositions and micro-morphologies of SSA particles,
  the chemical compositions and micro-morphologies of SSPs were much more uniform. In other
  words, grinding steel slag aggregates into SSPs can help homogenize the properties of steel slag.
  (2) The high alkalinity, rich convex and corrugated morphologies of the four SSPs improved the
  compatibility between filler particles and weak acid asphalt binder, which was beneficial for the
  stable bonding interaction between SSPs asphalt mastic and aggregate.
- 505 (3) The four SSPs can significantly improve the moisture stability, high-temperature stability and
   506 low-temperature crack resistance of asphalt mixture. SSP<sub>FF</sub> and SSP<sub>WF</sub> performed better in
   507 improving the moisture stability and high-temperature stability, while SSP<sub>FC</sub> and SSP<sub>WC</sub>
   508 performed better in enhancing the low-temperature crack resistance.

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