Additive

## 1 Performance of Zeolite Synthesized from Sewage Sludge Ash as a Warm Mix Asphalt

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Fuliao Zou<sup>a,1</sup>, Zhen Leng<sup>a,2\*</sup>, Ruijun Cao<sup>a,3</sup>, Gaoyang Li<sup>a,4</sup>, Yuan Zhang<sup>b,5</sup>, Anand Sreeram<sup>c,6</sup>

- <sup>6</sup> <sup>a</sup> Department of Civil and Environmental Engineering, The Hong Kong Polytechnic University,
- 7 Hung Hom, Kowloon, Hong Kong
- 8 <sup>b</sup> School of Civil Engineering and Transportation, South China University of Technology,
- 9 Guangzhou, Guangdong, China
- <sup>c</sup> Department of Civil, Architectural and Environmental Engineering, The University of Texas at
- 11 Austin, Austin, Texas, United States of America
- 12

# 13 Abstract

14 The disposal of sewage sludge ash (SSA) is a growing problem with both environmental and

- 15 economic ramifications. This study aims to synthesize zeolite from SSA as a foaming-based
- 16 warm mix asphalt (WMA) additive, and comprehensively characterize its performance. The
- 17 SSA-derived zeolite was first characterized using X-ray Diffractometer (XRD), Scanning
- 18 Electron Microscope (SEM), and Thermogravimetry-Differential Thermal Analyzer (TG-DTA)
- 19 tests. Then, WMA mixtures with the SSA-derived zeolite and a commercial zeolite additive, and
- 20 a conventional hot mix asphalt (HMA) mixture were produced in the laboratory for a wide range
- of engineering performance tests, including the indirect tensile stiffness modulus test, indirect
- 22 tensile fatigue test, moisture susceptibility test, and Hamburg wheel-tracking test. The
- experimental results indicated that the SSA-derived zeolite was pure zeolite A which can release
- approximately 20% crystal water gradually from 70 °C to 200 °C. The overall performance of the
- 25 WMA mixture with SSA-derived zeolite was better than that of the WMA mixture with
- commercial additive, and comparable to that of the HMA mixture except for rutting resistance,
- 27 which is marginally lower but still satisfactory. Cost-benefit analysis was conducted in the end,
- 28 which demonstrated that SSA-derived zeolite is eco-efficient. This study concluded that it is
- 29 feasible to use the SSA-derived zeolite as an effective and sustainable WMA additive.

30 Keywords: Sewage Sludge Ash, Zeolite Synthesis, Warm Mix Additive, Chemical Property,

31 Asphalt Mixture Performance, Cost Analysis

<sup>&</sup>lt;sup>1</sup> PhD Candidate, fu-liao.zou@connect.polyu.hk

<sup>&</sup>lt;sup>2\*</sup> Associate Professor, Tel: +852-2766-6007; Fax: 852-2334-6389; Email: zhen.leng@polyu.edu.hk

<sup>(\*</sup>Corresponding Author)

<sup>&</sup>lt;sup>3</sup> Post-Doctoral Fellow, ruijun.c.cao@connect.polyu.hk

<sup>&</sup>lt;sup>4</sup> PhD Student, 21064985r@connect.polyu.hk

<sup>&</sup>lt;sup>5</sup> Post-Doctoral Fellow, yuanzhang@scut.edu.cn

<sup>&</sup>lt;sup>6</sup> Post-Doctoral Fellow, anand.sreeram@austin.utexas.edu

### 32 1. Introduction

- 33 Warm mix asphalt (WMA) is a green paving technology that reduces the construction
- 34 temperature of asphalt mixtures without compromising their overall engineering performance
- 35 (Leng et al., 2014). Depending on the technology used in producing WMA, the construction
- temperature of the conventional hot mix asphalt (HMA) pavement can be lowered by about 20-
- 40 °C (Capitão et al., 2012; Rubio et al., 2012). This temperature reduction can be achieved by
- using organic additives, chemical additives, and foaming technologies (Kheradmand et al., 2014;
- 39 Xiao et al., 2011; Yu et al., 2018). In practice, foaming-based warm mix technology appears to
- 40 be preferred because of its lower cost and simple operation (Middleton and Forfylow, 2009). The
- foaming process can be accomplished by either direct water injection through a nozzle or by
- 42 adding foaming additives, such as zeolites containing crystal water in their micropore structure.
- 43 When zeolites are added into the asphalt mixture at construction temperatures, the crystal water
- 44 is released and vaporized, resulting in a rapid volume expansion or foaming of the asphalt binder,
- 45 which can improve the workability of asphalt mixtures and increase aggregate coating at lower
- 46 manufacturing temperatures. Both natural zeolites (e.g., clinoptilolite) and commercial synthetic
- 47 zeolites (e.g., Aspha-min, Advera) have been employed to produce WMA mixtures. It was
- 48 reported that the commercial synthetic zeolites can promote a better distribution of the binder
- 49 within the asphalt mixture and improve the compactability of asphalt mixtures without
- 50 negatively affecting the engineering performance (Kristjánsdóttir et al., 2007; Woszuk and
- 51 Franus, 2017). Natural zeolites can be considered as an alternative WMA additive to commercial
- 52 synthetic zeolites due to their additional economic and environmental benefits (Sengoz et al.,
- 53 2013; Valdes et al., 2018). Studies have also indicated that zeolites can be synthesized from
- 54 sewage sludge ash (SSA), a waste product commonly landfilled with growing environmental
- apprehension (Environmental Protection Department, 2020), which offers a potential value-
- added approach to recycle waste materials into WMA additives.
- 57 The management and disposal of sewage sludge from sewage treatment works has become a
- 58 global issue generating increasing environmental and economic concerns in high population
- 59 density cities around the world (Lam et al., 2015). Currently, sewage sludge is commonly
- 60 subjected to energy-intensive dewatering process and incineration, followed by disposal at local
- 61 landfill sites due to a lack of sustainable recycling approaches. With limited land resources and
- 62 the capacity of local recycling, there is a growing demand for novel approaches for the
- sustainable treatment of sewage sludge. Recent studies have investigated the utilization of SSA
  in cementitious materials and heavy metals adsorbent materials (Benassi et al., 2019; Wang et
- al., 2018a; Zhou et al., 2020). It was reported that the particle sizes of SSA can range from
- 66 submicron to around 700 µm (Cyr et al., 2007). The exact contents of the major elements and the
- amorphous glassy phases of SSA depend on the sludge treatment processes applied at
- 68 wastewater plants, as well as other factors such as industrial activities and the types of sewerage
- 69 system in the catchment area (Anderson and Skerratt, 2003; Wiebusch and Seyfried, 1997).

Nonetheless, the major components in SSA are Si, Al, Ca, Fe, P, and Na (Cyr et al., 2007; 70 71 Mahieux et al., 2010). Common crystalline forms of these elements are inert quartz (SiO<sub>2</sub>), 72 magnetite (Fe<sub>3</sub>O<sub>4</sub>), anorthoclase ((NaK)Al·Si<sub>3</sub>O<sub>8</sub>), and whitlockite (Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>) (Donatello and 73 Cheeseman, 2013; Zhang et al., 2018). It is interesting to note that synthetic zeolites may be produced from SSA, as zeolites are microporous hydrated aluminosilicates that mainly consist of 74 elements Al and Si (Cardoso et al., 2015; Kim and Lee, 2009). Among various types of zeolites, 75 Zeolite Linde Type A (Zeolite LTA or Zeolite A) presents beneficial commercial characteristics 76 that include thermal stability, high selectivity, and non-toxicity (Qian and Li, 2015). Moreover, it 77 can gradually release its crystalline water between 100 °C to 200 °C, which was found the most 78 suitable for being applied as a WMA additive (Afzal et al., 2000; Woszuk and Franus, 2017). 79 The general formula for zeolite A is Na<sub>12</sub>[(AlO<sub>2</sub>)<sub>12</sub>(SiO<sub>2</sub>)<sub>12</sub>] 27H<sub>2</sub>O wherein the framework 80 silicon to aluminum (Si/Al) ratio is close to one in most cases, and sodium ions are the 81 82 exchangeable extra-framework cations (Collins et al., 2020). The zeolite synthesis from loam minerals and waste materials containing Si and Al sources (e.g., kaolin, fly ash, and SSA) by the 83 hydrothermal reaction consists of three steps (Rios et al., 2009; Murayama et al., 2002): the 84 85 dissolution of Si and Al, the condensation of silicate and aluminate ions in alkali solution to make aluminosilicate gel, and the crystallization of aluminosilicate gel to make zeolite crystal. A 86 method was proposed to convert SSA to zeolite and apply it as a WMA additive for asphalt 87 pavement (Zhang et al., 2018). The alkaline fusion of SSA with the sodium hydroxide (NaOH), 88 followed by the hydrothermal reaction on the alkaline solution of the fused product was 89 conducted to synthesize the target zeolite. The produced SSA-derived zeolite was used as a 90 foaming additive for manufacturing the WMA mixture. The findings indicated that SSA-derived 91 zeolite has a high potential to be used as a WMA additive, and it has a similar temperature 92 reduction function as the commercial synthetic zeolite Aspha-min. However, the chemical 93 characteristics of SSA-derived zeolite and performance properties of the WMA mixture with 94 SSA-derived zeolite are still unknown. Since there are concerns about moisture susceptibility 95 and rutting resistance for WMA mixtures with zeolite additives (Hasan et al., 2015; Xu et al., 96 2017), it is necessary to comprehensively study the engineering performance of WMA mixtures 97 with SSA-derived zeolite for practical applications. Moreover, although the synthesis of SSA-98 99 derived zeolite removes SSA from landfilling, the economic efficiency of the synthesis process has not been evaluated. Therefore, the main objectives of this study are to investigate the 100 performance of the WMA mixture with SSA-derived zeolite through comprehensive laboratory 101 102 tests, compare the results with the WMA mixture prepared using Aspha-min and conventional HMA mixture, and characterize the cost-effectiveness of applying SSA-derived zeolite as a 103 104 WMA additive.

## 105 2. Research Materials and Methodology

## 106 2.1. Raw materials

- 107 Three types of mixes were prepared in this study, namely conventional HMA, warm mix asphalt
- 108 with SSA-derived zeolite (WMAZ), and warm mix asphalt with Aspha-min (WMAA). The
- 109 dosage of Aspha-min was 0.3% by the total mass of the asphalt mixture, which is recommended
- by the supplier and commonly applied in practice. The same dosage was used for the SSA-
- derived zeolite. All three mixtures shared the same mixture design, which was the polymer

- modified stone mastic asphalt with a nominal maximum aggregate size of 10 mm (PMSMA10),
- a surface course material for carriageway pavements commonly used in Hong Kong (Highways
- 114 Department, 2020). Styrene-Butadiene-Styrene (SBS) polymer modified bitumen that has a
- 115 Superpave performance grade of PG76-16 was used as the asphalt binder (AASHTO M320-10,
- 116 2013). The binder content was 6.0% by the total mass of the asphalt mixture. Local granite rocks
- 117 were used as the coarse and fine aggregates, as well as the mineral filler. Cellulose fiber and
- 118 hydrated lime were also used in the PMSMA10 mixtures according to local requirement. The
- cellulose fiber content was 0.3% by the total mass of the asphalt mixture. The gradation of
- 120 PMSMA10 is presented in **Table 1**.

Composition	Bulk specific gravity	Sieve size (mm)	Percentage (%)	
	2.642	14-10	3.5	
Coarse aggregates	2.663	10-5	59.5	
	2.709	5-2.36	9.0	
Fine aggregates	2.649	2.36-0.075	16.5	
Mineral filler	Aineral filler 2.661		9.5	
Hydrated lime	2.587	~0.075	2.0	

121 Table 1 Gradation of the PMSMA10 mixture

## 122 **2.2.** Synthesis of SSA-derived zeolite

SSA contains large percentages of silicon dioxide and aluminum oxide which can be used as source materials for the synthesis of zeolite. SSA-derived zeolite was synthesized based on the following procedure: SSA was first activated through the process of alkaline fusion using 500 g SSA and 575 g NaOH at 500 °C for 1 h. Then, the fused product was dissolved in the NaOH

- solution with a concentration of 2.5 mol/L at 65 °C for 3 h. Afterward, the sodium aluminate
- 128 (NaAlO<sub>2</sub>) powder was added to the supernatant solution to adjust the molar ratio of Si/Al to 0.8.
- 129 The product was crystallized at a temperature of 80 °C for 3 h and filtered out to obtain a white
- powder. Using this method, pure zeolite A, a suitable type of zeolite for WMA additives, can be

synthesized from SSA. The process to produce zeolite from SSA is illustrated in **Fig. 1**. The SSA

used to produce SSA-derived zeolite was provided by the sludge treatment facility (T-Park) in

133 Tuen Mun, Hong Kong.



### 135 Fig. 1. Synthesis of zeolite from SSA

### 136 **2.3.** Characterization of SSA-derived zeolite

- 137 X-ray Diffractometer (XRD) test, Scanning Electron Microscope (SEM) test, and
- 138 Thermogravimetry-Differential Thermal Analyzer (TG-DTA) test were conducted to
- 139 characterize the chemical and physical properties of the two zeolite-based WMA additives.
- 140 Rigaku SmartLab X-ray Diffractometer equipped with a 9-kW rotating anode X-ray source with
- 141 a high-quality semiconductor detector was used to identify the crystalline phases of the WMA
- additives. An X-ray wavelength ( $\lambda$ ) of 1.54 Å, a scanning step width of 0.02°, and a scanning
- speed of 4°/min were used in this study. Rigaku's PDXL software was used to compare the
- 144 detected XRD patterns with the standard patterns from the powder diffraction file (PDF)
- 145 database. Tescan VEGA3 SEM system that has a maximum magnification of 100,000X was used
- to characterize the morphology of WMA additives. Gold sputtering coatings were applied on the
- 147 WMA additives before the SEM tests. Rigaka Thermo plus EVO2 thermal analyzer was used to
- determine the thermal properties of WMA additives. During the TG-DTA tests, the WMA
- additives were heated from 25 °C to 500 °C at a heating rate of 10 °C/ min.

## 150 **2.4. Mixture specimen preparation**

- 151 The HMA mixture was mixed at 180 °C and compacted at 160 °C. The WMA mixtures were
- mixed at 155 °C and compacted at 135 °C. Superpave Gyratory Compactor (SGC) was used to
- produce test specimens. The air void contents of the produced specimens were  $4\% \pm 0.5\%$  for the
- 154 Indirect Tensile Stiffness Modulus (ITSM) and Indirect Tensile Fatigue (ITF) tests. The air void
- 155 contents of the produced specimens were  $7\% \pm 0.5\%$  for the moisture susceptibility test and the
- 156 Hamburg wheel-tracking test. Three replicate specimens were prepared and tested for each test
- 157 except for the Hamburg wheel-tracking test, which used two replicate specimens. The general
- information on the tests and the prepared specimens is summarized in **Table 2**.

## 159 **2.5. Laboratory tests for engineering performances**

- 160 Asphalt pavements are prone to surface distresses such as fatigue cracking, moisture damage,
- rutting, and thermal cracking under heavy traffic loading (Jiang et al., 2020; Li et al., 2020;

- 162 Wang et al., 2018b). Since the local climate in Hong Kong is relatively hot and humid, this study
- 163 focused on the intermediate temperature durability and high temperature performance of the
- 164 produced asphalt mixtures. The ITSM and ITF tests were conducted to investigate the cracking
- resistance of the produced asphalt mixtures at intermediate temperatures. The freeze-thaw cycle
- 166 conditioned moisture susceptibility test was conducted to evaluate the moisture susceptibility of
- 167 the produced asphalt mixtures. The immersion Hamburg wheel-tracking test was conducted to
- 168 investigate the rutting resistance of the produced asphalt mixtures under high temperature and
- 169 humidity.

### 170 2.5.1. ITSM and ITF tests

- 171 Test specimens with a diameter of 100mm and a thickness of 40mm were produced for the ITSM
- test and ITF test, according to BS DD 213 (1993) and BS EN 12697-24 (2012), respectively. The
- 173 ITSM test was conducted on both unaged and aged specimens to evaluate the differences in
- stiffness modulus before and after aging. The aged specimens were subsequently used to run the
- 175 ITF test. The ITF test was conducted under the stress-controlled mode at three stress levels. The
- aged specimens were produced according to AASHTO R30-02 (2010), in which the loose
- 177 mixtures were conditioned in a force-draft oven for 4 h at 135 °C for short-term aging and then
- 178 compacted and aged in the oven for 5 days at 85 °C.

## 179 **2.5.2.** Moisture susceptibility test

- Test specimens with a diameter of 100mm and a thickness of 62.5mm were produced for the moisture susceptibility test, according to ASTM D4867-09 (2014). The Tensile Strength Ratio (TSR) between the mixtures with the freeze-thaw cycle conditioning and the mixtures without the freeze-thaw cycle conditioning was measured to evaluate the moisture susceptibility. The specimens conditioned with freeze-thaw cycle were partially saturated with water and conditioned in a freezer at -18 °C for 16 h and then in a water bath at 60 °C for 24 h. The
- 186 Dynamic Testing System (DTS-30) from PAVETEST was used for measuring the ITSM, ITF,
- and TSR.

# 188 2.5.3. Hamburg wheel-tracking test

- 189 Test specimens with a diameter of 150mm and a thickness of 60mm were produced for the
- 190 Hamburg wheel-tracking test, according to AASHTO T324-11 (2013). The Immersion Wheel
- 191 Tracker (CRT-WTIM) from Cooper Research Technology equipped with a moving steel wheel,
- a water bath, and a linear variable differential transducer system for measuring the rut depth was
- 193 used for this test. During the test, the specimens were submerged in water and subjected to a
- steel wheel loading with a rolling speed of 52 passes/min. The water temperature can be set from
- 195 25 °C to 70 °C, with 50 °C being the most common test temperature, which was used in this
- study. Except for running the test at 50 °C, a water temperature of 60 °C was also chosen in this

- study to investigate the high temperature performance of produced HMA and WMA mixtures. 197
- Rut depth was measured at every 200 passes of the wheel and a maximum of 20,000 passes was 198
- applied for each test. 199

Test	Mixture	Mixing/ compaction temperature (°c)	Specimen diameter/ thickness (mm)	Air void content (%)	Test temperature (°c)	Condition
Indirect tensile	HMA	180/160		3.5 - 4.5	20 ± 0.2	Unaged & aged
stiffness	WMAZ	155/135	100/ 40			
modulus	WMAA	- 155/ 155				
Indirect tensile fatigue	HMA	180/160	100/ 40	3.5 - 4.5	20 ± 0.2	Aged
	WMAZ	- 155/ 135				
	WMAA					
Moisturo	HMA	180/160				
susceptibility	WMAZ	- 155/ 135	100/ 62.5	6.5 - 7.5	$25\pm0.5$	Unaged
	WMAA					
Homburg	HMA	180/ 160	150/ 60	6.5 - 7.5	$50 \pm 1.0$ &	Unaged
wheel-tracking	WMAZ	- 155/ 135				
	WMAA				00 ± 1.0	

200 Table 2 General information of the tests and specimens

#### 2.6. Cost-benefit analysis 201

202 After characterizing the engineering performance of the WMA mixture prepared with SSAderived zeolite, a cost-benefit analysis was conducted to quantify the cost-effectiveness of the 203 204 synthesis process of SSA-derived zeolite. It is worth noting that producing 1 kg zeolite would also generate 1.18kg mineral filler as a by-product, which could replace part of the filler required 205 for preparing asphalt mixture. Therefore, in the cost-benefit analysis, the cost mainly covered the 206 input material and energy cost, and the benefit was considered as the saving brought by the by-207 product from the synthesis process. Table 3 lists the unit cost and quantity of the input items that 208 209 produced 1kg zeolite. The energy use was evaluated as the electricity consumption measured in the lab during the synthesis treatment. The material cost was collected from the Chinese 210 manufacturer suppliers, and the electricity cost was obtained from the China Light and Power Co 211 Ltd based on the current energy structure in Hong Kong. The monetary values of all items have 212 been converted to equivalent US dollars. For the benefit from the by-product, the cost evaluation 213 result would largely depend on the corresponding filler type and the content it replaced. 214

- 215 According to the survey in relevant Chinese manufacturer suppliers, the unit cost of mineral
- filler ranged from \$0.02 to \$0.1 per kilogram depending on the varied recipes.

Input items		Unit cost (\$)	Quantity
	SSA	NA	1.53kg
Material	NaOH	0.01	0.14kg
	NaAlO <sub>2</sub>	0.36	0.51kg
Energy	Electricity	0.16	3.11KWh

Table 3 Cost inventory of the synthesis process derived from SSA

### 218 **3. Results and Discussion**

## 219 **3.1. Characterization of SSA-derived zeolite**

Fig. 2. shows the XRD patterns of SSA-derived zeolite and Aspha-min. It can be observed that

221 SSA-derived zeolite had a XRD pattern very similar to that of the Aspha-min. Only a few

differences in the peak locations with very low intensity can be found in the patterns, suggesting

that SSA-derived zeolite had a very similar crystalline structure compared with Aspha-min.

224 There were minor differences in the peak intensities, which indicates the grain size of these two

225 materials are different. Comparing their XRD patterns with the standard patterns from the

powder diffraction file database supplied by the International Centre for Diffraction Data

227 (ICDD), it was found that only the crystalline phase of zeolite A was included in the SSA-

derived zeolite. In Aspha-min, the major crystalline phase was also zeolite A. But some other

229 unknown crystalline phases were also observed. Zeolite A has a three-dimensional pore structure

230 with pores running perpendicular to each other in the x, y, and z planes. The pore diameter is

defined by an eight-member oxygen ring and is small at 4.2 Å, which leads to a larger cavity of a

232 minimum free diameter of 11.4 Å.



Fig. 2. XRD patterns of: (a) SSA-derived zeolite; and (b) Aspha-min 235

Fig. 3. shows the SEM images of the SSA-derived zeolite and Aspha-min. It is clear that 236 both materials had a simple cubic crystalline structure. The difference between SSA-derived 237 zeolite and Aspha-min was their grain size wherein SSA-derived zeolite had a smaller grain size. 238 239 The SSA-derived zeolite had a crystalline size of about 1µm in diameter while the Aspha-min had a crystalline size of about 2µm in diameter. The chemical compositions of SSA-derived 240 zeolite and Asphamin were determined by X-ray fluorescence (XRF) analysis. Table 4 presents 241 their chemical compositions in a form of major elements at weight percentages. The thermal 242 properties of SSA-derived zeolite and Aspha-min are illustrated in Fig. 4. The weight loss over 243 the range 25-200 °C confirms the release of water and the weight loss over the range 200-500 °C 244 indicates the thermal decomposition and the release of the pyrolysis products (de Angelis Curtis 245 et al., 1999; Duan et al., 2008; Saldo et al., 2002). It can be observed that both the SSA-derived 246 zeolite and Aspha-min released crystal water when the temperature was increased, but Aspha-247 min had less weight loss and an earlier heat absorption peak than the SSA-derived zeolite. 248 Aspha-min showed a weight loss of 20.5% while the observed weight loss from SSA-derived 249 zeolite was 23.4%, which was 14% larger than that of the Aspha-min. The heat absorption peak 250 was at 113 °C for Aspha-min while it was at 136 °C for the SSA-derived zeolite. The results 251 indicated that SSA-derived zeolite can release more crystal water and the release is steadier when 252

heated. This is because SSA-derived zeolite has a higher Si/Al ratio (1.28) compared to 253 Asphamin (1.19) (see **Table 4**). The water-binding force of zeolite is increased with the higher 254 Si/Al ratio, which results in slower water release (Woszuk et al., 2017). Also, divalent cations 255 (e.g.,  $Mg^{2+}$  and  $Ca^{2+}$ ) are more hydrated by water particles than monovalent cations. SSA-256 derived zeolite has a total amount of 0.78 wt % divalent cations, which is more than double 257 compared to that of Asphamin (0.36 wt %). Moreover, SEM results confirmed the smaller grain 258 size of SSA-derived zeolite compared to Asphamin, which results in a larger specific surface 259 area and volume that contribute to a higher number of water-binding sites per mass unit. These 260 characteristics determined the higher water binding ability and stronger water-binding force of 261 SSA-derived zeolite, which results in an expected better foaming effect and asphalt coating of 262 WMA mixtures with SSA-derived zeolite. Both the SSA-derived zeolite and Aspha-min started 263 to release crystal water when heating begins, and the release became more significant at around 264 265 80 °C and continued up to approximately 190 °C, then, SSA-derived zeolite and Aspha-min slowly decomposed as temperature rises from 190 °C to 500 °C. The observed percentage weight 266

- losses of crystal water from both the SSA-derived zeolite and Aspha-min were approximately
- 268 20%.



Fig. 3. SEM images of SSA-derived zeolite ((a) 10k× and (b) 20k×) and Aspha-min ((c) 10k× and (d)  $20k\times$ ) 

Table 4 XRF result of SSA-derived zeolite and Asphamin illustrating the quantities (wt %) of 

major elements. 

Component	SSA-derived Zeolite (wt %)	Asphamin (wt%)
Na	20.00	22.60
Mg	0.26	0.27
Al	33.80	32.40
Si	43.10	38.60
Р	0.12	0.08
S	0.17	5.15
Cl	0.08	0.48
Κ	1.15	0.23
Ca	0.52	0.08
Fe	0.71	0.07





Fig. 4. (a) weight loss and (b) heat flow of SSA-derived zeolite and Aspha-min 278

#### 279 3.2. Indirect tensile stiffness modulus of HMA and WMA mixtures

The ITSM test results are shown in Fig. 5. The indirect stiffness moduli of unaged HMA, 280

WMAZ, and WMAA were 2,371 MPa, 2,018 MPa, and 2,148 MPa, respectively. It can be 281

observed that the WMA mixtures showed lower values in stiffness modulus than the HMA 282

mixture before aging. This was expected because the WMA mixtures had undergone 283

284 considerably lower aging during manufacturing due to the 25 °C lower production temperature.

- The WMAZ mixture showed the lowest stiffness modulus that was 15% lower than that of the 285
- HMA mixture and 6% lower than that of the WMAA mixture, which is helpful for cracking 286
- resistance at intermediate temperatures. After aging, the stiffness moduli of the WMA mixtures 287
- were slightly lower than the HMA mixture, but no significant difference was observed. The 288
- 289 indirect stiffness moduli of aged HMA, WMAZ, and WMAA were 3,398 MPa, 3,300 MPa, and
- 3,367 MPa, respectively. The HMA mixture had the highest stiffness modulus which was 3% 290

- higher than the WMAZ mixture and 1% higher than the WMAA mixture. This indicated that the 291 lowered manufacturing temperature of WMA mixtures provides insignificant effects on their 292 long-term cracking resistance. It was noticed that the WMAA has a slightly higher stiffness 293 294 modulus compared to the WMAZ in both unaged and aged states. The filler effect may explain the discrepancies in their stiffness moduli. As both SSA-derived zeolite and Asphamin are 295 296 crystalline microporous hydrated aluminosilicates, after releasing the crystal water, residual dehydrated aluminosilicates stay in the asphalt mixture and act as fillers. It was found that 297 Asphamin has lower crystal water content than SSA-derived zeolite. Thus, when using the same 298 application dosage, Asphamin was expected to have a higher number of fine aluminosilicates 299 that stayed in the asphalt mixture as fillers after foaming completed, which results in the slightly 300 higher stiffness modulus of the WMAA compared to the WMAZ (Abbas et al., 2005; Baskara et 301 302 al., 2019). Moreover, it was found that asphalt mixtures may exhibit lower stiffness modulus
- 303 when finer fillers are used (Muniandy and Aburkaba, 2011). This may be another reason that the
- 304 WMAZ presents a lower stiffness modulus compared to the WMAA as the grain size of SSA-
- 305 derived zeolite is smaller than Asphamin.







### 308 **3.3. Fatigue performance of HMA and WMA mixtures**

- 309 The fatigue tests were conducted over an initial tensile strain range of 100  $\mu\epsilon$  to 400  $\mu\epsilon$ , and the
- resultant fatigue life of the tested material shall fall within a range between  $10^3$  and  $10^6$  per
- number of load applications. The conventional failure criterion  $N_{f/50}$  was employed for
- estimating the fatigue life of the test specimens. Fig. 6. shows the fatigue life versus the initial
- 313 strain of the HMA and WMA mixtures. The least-squares regression relationship was fitted to

- 314 the data of the initial strain as an independent variable and the data of the number of load 315 applications of the fracture life as a dependent variable according to Equation 1:
- 316  $N_f = k \times \left(\frac{1}{\epsilon_0}\right)^n$  (1)

where N<sub>f</sub> is the number of load applications until fracture; k, n are material constants; and  $\varepsilon_0$  is the initial tensile strain in  $\mu\varepsilon$ .

It can be observed that the HMA mixture and WMA mixtures had very similar fatigue lives in 319 the log-scale diagram. The results suggested that the fatigue performances of the three mixtures 320 are very close to each other. The fatigue lives of the WMA mixtures were slightly lower than the 321 HMA mixture in high strains. Among the three mixtures, the WMAA mixture had the lowest and 322 323 the HMA mixture had the highest fatigue life in high strains. There was no obvious difference in the fatigue life of the HMA mixture and the WMA mixtures in middle strains. In low strains, the 324 WMAA mixture showed the highest fatigue life. Table 5 presents the parameters of the 325 regressional fatigue lines, as well as the predicted fatigue lives corresponding to the strains of 326 100  $\mu\epsilon$ , 200  $\mu\epsilon$ , and 400  $\mu\epsilon$ , respectively. The predicted fatigue lives corresponding to the high 327 stain (400 µɛ) for HMA, WMAZ, and WMAA were 3,170, 3,044, and 2,878, respectively. This 328 suggested that the HMA mixture has better resistance to heavy good vehicles than the WMA 329 330 mixtures. The predicted fatigue lives corresponding to the middle strain (200 µɛ) for both WMA mixtures were 2% lower than that of the HMA mixture. The predicted fatigue life corresponding 331 to the low strain (100 µɛ) for the WMAA mixture was 526,045, which was 6% higher than those 332 of the other two mixtures. The results indicated that WMA mixtures have slightly lower fatigue 333 resistance at high stain levels and slightly better fatigue resistance at low strain levels. But 334 335 overall, the fatigue resistances of the three mixtures are close to each other.



337 Fig. 6. Fatigue life versus initial strain for HMA and WMA mixtures

Parameter				Predicted fatigue life		
Mixture	ixture k n	$\mathbf{P}^2$	cycles @	cycles @	cycles @	
	K	11	K	100 με	200 με	400 με
HMA	9.790E+12	3.647	0.990	497,488	39,712	3,170
WMAZ	1.105E+13	3.674	0.943	495,864	38,849	3,044
WMAA	1.718E+13	3.757	0.981	526,045	38,909	2,878

Table 5 Parameters of the fatigue lines and the predicted fatigue lives at different strains

### 339 3.4. Susceptibility to moisture damage of HMA and WMA mixtures

340 The effects of moisture on the WMA mixtures were investigated by measuring the tensile

341 strength ratio between the mixtures with the freeze-thaw cycle conditioning, and the mixtures

342 without the freeze-thaw cycle conditioning. The moisture susceptibility of the HMA mixture was

also investigated for comparison purposes. Fig. 7. shows the results of the moisture susceptibility

- tests. The results indicated that the WMA mixtures had slightly higher strengths than the HMA
- mixture. The dry strengths of the HMA, WMAZ, and WMAA were 9.6 kN, 9.8kN, and 9.7 kN,
- 346 respectively. After the freeze-thaw conditioning, there was an obvious decrease in the indirect
- tensile strength for all mixtures. The wet strengths of the HMA, WMAZ, and WMAA were 7.8

kN, 8.1 kN, and 8.0 kN, respectively. It can be observed that the WMAZ mixture had the highest

349 strength both before and after conditioning, but the difference is not significant. The tensile

strength ratios for HMA, WMAZ, and WMAA were 80.87%, 82.71%, and 82.14%, respectively.

- 351 The results suggested that there is no specific concern about the moisture susceptibility of WMA
- mixtures prepared by using SSA-derived zeolite as the additive. The usage of lime might be one
- 353 of the main contributing factors to the satisfactory moisture damage resistance of all the
- 354 mixtures. The discrepancies in the micro-properties of SSA-derived zeolite and commercial
- zeolite Asphamin may be the reason that leads to the higher tensile strengths of the WMAZ both
- dry and wet compared to the WMAA. It was found that SSA-derived zeolite has a higher Si/Al
- ratio, more divalent cations, and smaller gran sizes compared to Asphamin, which results in
- 358 higher water binding ability and stronger water-binding force of SSA-derived zeolite. It was
- 359 observed that SSA-derived zeolite can release water more gradually, which results in a better
- 360 foaming effect. Therefore, the better foaming effect and aggregate coating improved the strength
- development of the WMAZ compared to the WMAA.





### 364 **3.5. Rutting resistance of HMA and WMA mixtures**

362

From the Hamburg wheel-tracking test, the following three performance parameters of the testing specimens are commonly obtained: a) creep slope: the depth of rutting per loading pass in the creep stage, which indicates the rutting resistance; b) stripping slope: the depth of rutting per loading pass in the stripping stage, which indicates the resistance to moisture damage; and c) stripping inflection point (SIP): the number of passes at which the regression lines of the creep stage and the stripping stage intersects, indicating the start of moisture damage.

- Fig. 8. shows the results of the Hamburg wheel-tracking tests of HMA and WMA mixtures at
- two different temperatures. The values corresponding to the creep slope and the maximum rut
- depth are given in **Table 6**. It was noticed that the stripping slope and the SIP were not observed

- in the results, indicating the good moisture damage resistance potential of the testing mixtures,
- which is consistent with the results of the moisture susceptibility tests. From **Fig. 8** (a)., it can be
- seen that the HMA mixture had the lowest rut depth of 3.20 mm after 20,000 cycles of loading at
- 50 °C, while the values for the WMA mixtures were higher and the WMAA mixture had the
- highest rut depth of 3.92 mm. The results indicated that the HMA mixture provided better rutting
- resistance than the WMA mixtures. This was expected because the production temperatures of
- 380 WMA mixtures were considerably lower than the HMA mixture. The rutting potential increases
- 381 with the decrease of mixture production temperatures, which is related to the decreased aging of
- asphalt binder in the WMA mixtures (Bennert et al., 2011). The results also suggested that the
- 383 WMAZ mixture had better rutting resistance than the WMAA mixture. The maximum rut depth
- of the WMAZ mixture was 5% lower than that of the WMAA mixture. For asphalt mixtures with
- a PG-76 binder, it is common to allow for a maximum rut depth of 12.7mm (0.5 in.) at 20,000
- passes in Hamburg wheel-tracking test (Yildirim et al., 2007). Thus, it can be noted that the
- 387 measured wheel-tracking depths of both WMA mixtures satisfied the recommended requirement,
- although they were slightly larger than that of the HMA mixture.
- 389 To understand their rutting resistances in a harsher environment, the Hamburg wheel-tracking rut
- depths of the three mixtures were also measured at a higher temperature of 60 °C, as presented in
- **Fig. 8 (b)**. It is obvious that the rut depths of all mixtures were significantly increased due to the
- increased testing temperature, but the ranking followed the same trend as that at 50 °C. It was
- also interesting to notice that even though the test temperature had been increased by  $10 \,^{\circ}$ C, the
- maximum rut depths of the three mixtures were still smaller than 12.7 mm. These results
- suggested that all the three mixtures had good rutting resistance even in the case of extremely hot
- 396 weather.





Fig. 8. Hamburg wheel-tracking curves of HMA and WMA mixtures at: (a) 50 °C; and (b) 60 °C

Minturo	Test temperature	Creep slope	Maximum rut depth	
Mixture	(°C)	(mm/ 1000 passes)	(mm)	
HMA		0.061	3.20	
WMAZ	50	0.078	3.74	
WMAA		0.086	3.92	
HMA		0.269	8.06	
WMAZ	60	0.314	9.33	
WMAA		0.344	9.89	

400 Table 6 Summary of the values of creep slope and the maximum rut depth

### 401 **3.6. Cost-benefit analysis**

Fig. 9. illustrates the evaluated cost-benefit results for producing 1 kg SSA-derived zeolite. 402 Based on the cost inventory, the total cost for the recycling process is \$0.87/kg. The advantage in 403 the economy of the SSA recycling could be observed, compared with the cost of \$1.35/kg for 404 405 commercial zeolite additive (Sukhija and Saboo, 2020). The large proportion of NaAlO<sub>2</sub> enables the price of NaAlO<sub>2</sub> to become the most influential factor in the overall synthesis cost. In 406 407 addition, due to the varied costs of mineral filler, the benefit from the by-product ranged from \$0.02-\$0.12 when producing 1 kg zeolite. As the fixed cost of SSA-derived zeolite is calculated 408 based on the market price of the raw materials and energy consumption collected under the 409 laboratory conditions, the cost is expected to be reduced under the mass production when facing 410 the market. Although this evaluation may be lower than its potential market price as it did not 411 consider the rate of return, it provides the initial insight into the benefit of cost-efficiency of 412

413 SSA-derived zeolite.





### 416 4. Summary and Findings

414

In this study, synthetic zeolite was first derived from SSA and its chemical properties were characterized and compared with those of the commercial WMA additive, Aspha-min. Then, two WMA mixtures (WMAZ & WMAA) were produced by using SSA-derived zeolite and Asphamin as additives, respectively. Finally, comprehensive laboratory tests were conducted to study the performance properties of the produced WMA mixtures in comparison with an HMA mixture with the same gradation, followed by a cost-benefit analysis. Based on the outcomes of this study, the following findings were obtained:

- The SSA-derived zeolite was characterized to be pure zeolite A which can release
- 425 approximately 20% crystal water gradually from 70 °C to 200 °C. It has a similar crystalline
- 426 structure as the commercial zeolite, Aspha-min, but with a smaller grain size. Compared with

- 427 Aspha-min, the SSA-derived zeolite contained a relatively larger percentage of crystal water428 and provided a more gradual release of crystal water when heated.
- The stiffness modulus of the WMAZ mixture was 15% lower than the HMA mixture and 6% lower than the WMAA mixture. Such differences in the stiffness modulus became very subtle after aging. The produced HMA and WMA mixtures showed very similar resistances to fatigue cracking, and there was no significant difference observed in their fatigue lives.
- The WMAZ mixture showed slightly higher strengths compared to the HMA and WMAA mixtures. The tensile strength ratios for WMAZ, HMA, and WMAA were 82.71%, 80.87%, and 82.14%, respectively, indicating similar good resistance to moisture damage of all mixtures.
- The rutting resistances of all three mixtures were satisfactory, although the two WMA
  mixtures showed larger rut depth than the HMA mixture, which was mainly caused by the
  decreased aging of the WMA mixtures during production. Compared with WMAA, WMAZ
  showed slightly better rutting resistance.
- SSA-derived zeolite can reduce the 35.6%-50% of the cost per kilogram, which shows its advantages in the economy compared with commercial zeolite products based on the data collected in this study.
- 444 To summarize, the overall engineering performance of the WMA mixture with SSA-derived zeolite additive was similar to the HMA mixture and superior to the WMA mixture with the 445 commercial foaming additive in terms of cracking resistance, fatigue resistance, and moisture 446 447 susceptibility. It is anticipated that the utilization of such SSA-derived zeolite materials could open a new outlet for the economical disposal of waste products currently landfilled and help 448 relieve the strain of disposal. However, it should be noted that the findings of this study were 449 450 based on the results of laboratory tests on one type of asphalt mixture. Laboratory tests on different asphalt mixtures and field trials are recommended in the future to further verify the 451 452 performance of the SSA-derived zeolite as a WMA additive.

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