

Stone Matrix Asphalt Performance with Glycerin Pitch as Asphalt Binder Extender

Atmy Verani Rouly Sihombing^{1*}, Retno Utami¹, Andri Krisnandi Somantri¹, Aditia Febriansya¹, Reza Phalevi Sihombing², Agah Muhammad Mulyadi³

¹ Department of Civil Engineering, Bandung State Polytechnic, Bandung 40559, Indonesia

² Department of Architecture, Bandung National Institute of Technology, Bandung 40124, Indonesia

³ Regional Planning, Research, and Development Agency of Cimahi City, Cimahi 40513, Indonesia

* Corresponding author, e-mail: atmyvera@polban.ac.id

Received: 08 January 2023, Accepted: 17 February 2023, Published online: 02 March 2023

Abstract

This research aims to utilize glycerin pitch waste as an alternative asphalt binder (extender) in Stone Mastic Asphalt (SMA) mixture. Glycerin pitch (GP) is a by-product from crude glycerol refining process from the palm oil oleochemical industry. The research method was carried out by testing the chemical and physical characteristics of GP by comparing it to pen 60/70, then testing based on asphalt binder rheology and asphalt concrete characteristics. ANOVA statistical test and Tukey-Kramer statistical grouping test were used to know the significant use of GP in SMA mixture. The variations of GP content tested to pen 60/70 were 0%, 15% and 25% by weight, then used as a binder for the SMA mixture which was tested under asphalt content (Pb) conditions based on Marshall characteristic, immersion index and drain down. The test results show that GP has the potential as an asphalt extender, with a maximum content of 22% by weight of asphalt binder or with a ratio of 1 GP: 4 pen 60/70. The characteristics of the SMA mixture using pen 60/70 and GP as asphalt binder extender, under conditions of Pb = 7% produce SMA mixture that meets the specification. Statistically, in stability characteristics of SMA, GP has a significant effect on SMA + 22% GP mixture. In the immersion index, the use of GP has a significant effect on all type of mixture, while in the draindown characteristics has significant effect on the SMA+0% GP mixture.

Keywords

oleochemical, glycerin pitch, stone matrix asphalt, asphalt binder extender, pen 60/70

1 Introduction

Stone Matrix Asphalt (SMA) is a type of asphalt concrete with 70% to 80% coarse aggregate and 11% filler [1, 2]. SMA mixtures are more resilient to deformation, has a higher skid resistance due to high coarse aggregate content. SMA also tends to last longer in time due to high asphalt content and stabilized with cellulose fiber can perform better under heavy vehicles [3].

The number of usages of asphalt as binder in SMA mixtures is at 5.5% to 7% and is considered high number of contents, compared to other type of asphalt mixtures [4–7]. This is because SMA is hoped to produce a pavement with higher skid resistance. Due to high asphalt content in the SMA mixture the application of the pavement is still limited in Indonesia, only in road or highway with high-speed vehicles that need safety factor, such as racing circuit, aircraft runway, toll road and others.

In the future, the existence of roads or highway is not only required to provide comfort to road users, but also safety. SMA is a mixed type that can meet the needs of road users for the comfort and safety of road pavements [3]. To overcome this, the government needs to prepare from now on, one of which is preparing for the need for more asphalt binder use, it's just that up to now, Indonesia still relies on imports to meet 83% of its asphalt binder needs for road construction [8]. One way to overcome this problem is to replace some of the asphalt binder needs with asphalt binder substitutes/extenders.

Asphalt binder extender is an asphalt-like material that can be used to substitute or partially replace asphalt as a binder without reducing the performance of the asphalt concrete [9, 10]. Finding replacement materials for asphalt or improving the service performance of pavement received significant relevance from the life cycle

assessment (LCA) standpoint [11, 12]. As a country with enormous natural resources, Indonesia has many sources that can produce oil and its derivatives, one of which is the oleochemical palm oil industry. This oleochemical industry has existed in Indonesia since 1975, the main product of this industry is palm oil which of course produces side materials, or which are considered waste or by-product because they cannot be reused. It's the same as petroleum processing which produces tar/asphalt as a side material which was previously considered valueless, but with the development of research on asphalt is used as a binder for asphalt concrete mixtures. Utilization of waste from the oleochemical industry is also expected to be like asphalt which can be developed so that the waste has more value.

One of the products from the oleochemical industry is glycerol, which is widely used in industries such as pharmaceuticals, medical, food, and so on [13]. Annually, Indonesia produces 650,000 tons of glycerol which in the refining process will produce a by-product material called glycerin pitch or GP as much as 3% of the total production [14, 15]. An industry that produces waste is of course required to provide a waste treatment plant in order to protect the environment. Since 2014, the oleochemical industry has only had 9 (nine) waste treatment plants with a processing capacity of 1.40 million tons/year [16]. In the future the number of these factories will not be able to handle the waste processing from oleochemical production in Indonesia, namely as much as 23.3 million tons/year, which currently has the largest palm oil-based oleochemical capacity in the world [17].

Glycerin pitch (GP) is a dark brown viscous liquid that contains non-glycerol organic compounds and has a high mineral content [18]. Physically, this brownish-black GP has a gel-like viscosity similar to asphalt/tar/bitumen. From previous research, GP was used as binder to replace asphalt in producing asphalt roof shingles (ARS) which comply with the minimum requirements for roof tile strength based on ASTM C 1492-03 [19] and water absorption according to ASTM C 1167-03 [20]. ARS is a tile made of bitumen as a binder and is commonly used in several countries

because it has advantages such as being durable, affordable, and suitable for use in countries with low temperatures. Dry strength glycerin pitch roof shingles (GPRS) value of 5385.40 N and a wet strength of 1139.96 N, and absorption with a value of 4.66% were obtained [21]. Based on this, the physical characteristics of GP are similar to asphalt, making the reason for using GP as a binder in the manufacture of asphalt mixtures for road pavements.

In this research, the utilization of GP as a substitute for asphalt for Stone Mastic Asphalt (SMA) mixture is expected to be the initial stage of utilization of oleochemical waste in Indonesia. The test to determine GP characteristic needs to be carried out first, then further test is carried out to see its effect on oil asphalt and how it affects SMA mixture, so then GP is hoped can be use as asphalt substitution.

2 Materials and method

2.1 Materials

The materials used in this research are:

- Asphalt pen 60/70, was chosen because according to the Highway General Specification of Indonesia standard, 2018 [22], the use of a binder for the Stone Mastic Asphalt mixture is asphalt with criteria according to the specifications for asphalt pen 60/70 [22].
- Glycerin Pitch (GP) originating from the palm oil oleochemical industry in Bekasi Regency, West Java Province, Indonesia which is produced from the glycerol purification process by the hydrolysis route as shown in Fig. 1.

2.2 Test method

The characteristics of glycerin pitch (GP) were carried out by testing the characteristics, chemical structure, saponification rate test [23], organic content [24], and the compounds contained in the GP. Chemical structure testing is carried out using the FTIR (Fourier Transform Infrared Spectroscopy) device while testing the GP compounds is tested using XRD and GCMS (Gas Chromatography and Mass Spectroscopy), on asphalt this test can identify the number of carbon (C) atoms [25, 26].

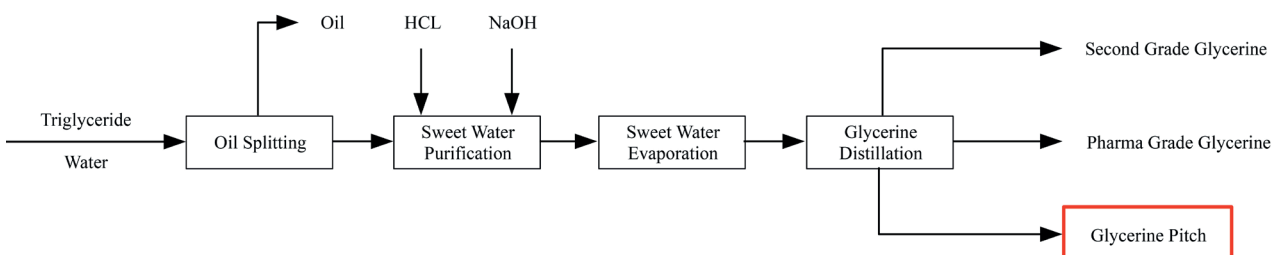


Fig. 1 Hydrolysis route glycerol purification process

Number of C atoms test is carried out to identify which distillate material is classified in which fuel category, for example in the distillation of petroleum in the form of binder asphalt it has a number of C atoms > 20, if it is in the form of a lubricant it has a number of C atoms of 15 and the smaller the number in line with lower temperatures in the petroleum distillation process, which at temperatures < 25 °C the results of petroleum distillation are in the form of gas [27]. In addition, the further test is carried to classified whether GP waste is as organic or non-organic material, even though GP is produced from organic material, but has gone through various glycerol purification processes. By knowing the characteristics of GP, it can then be considered for drawing conclusions on the results of studies regarding the influence of organic or inorganic materials on the characteristics of asphalt and asphalt mixtures.

The method used in this research was adding glycerin pitch to asphalt pen 60/70 with variation of GP content of 0%, 15%, 20%, 25%, and 30% to the weight of asphalt pen 60/70 mixed using a hot plate magnetic stirrer device as shown in Fig. 2 at a temperature of 120 °C for 15–20 minutes with a mixing speed of 0.4–0.6 kr/sec [28] until homogeneous [29–31]. Once homogeneous, the asphalt resulting from mixing GP and asphalt pen 60/70 is tested based on the basic asphalt test that has been used since the 19th century [32], namely penetration and softening point [33, 34].

The maximum GP content which still produces asphalt with asphalt pen characteristics of 60/70, was then tested for its chemical characteristics using Fourier-transform infrared spectroscopy (FTIR) [35] and asphalt rheology tests.

The FTIR-Alpha-P from Bruker Optics Company, which was controlled by the opus software, had a resolution of 4 cm⁻¹, a scanning speed of 32 sheets per second, and a chosen frequency range of 4000–500 cm⁻¹. For additional investigation, functional groups were established and contrasted. In addition to the IR spectra, changes in

the carbonyl group (C=O) and sulfoxide group (S=O) can also be used to determine whether an asphalt binder has been modified [36–38]. Carbonyl (-C=O) is represented by the band at 1700 cm⁻¹ [39] and sulfoxide is represented by the band at 1030 cm⁻¹. Using the equation presented by Lamontagne et al. [40], the analysis was conducted by assessing the carbonyl index (I_c) and the sulfoxide index (I_s), as shown in Eq. (1) and Eq. (2).

$$I_{c=O} = \frac{\text{Area of the carbonyl band around } 1.700 \text{ cm}^{-1}}{\text{Area of the spectral bands between } 2.000 \text{ and } 600 \text{ cm}^{-1}} \quad (1)$$

$$I_{s=O} = \frac{\text{Area of the sulfoxide band around } 1.030 \text{ cm}^{-1}}{\text{Area of the spectral bands between } 2.000 \text{ and } 600 \text{ cm}^{-1}} \quad (2)$$

Binder asphalt rheology tested under unaged asphalt conditions and RTFO (rolling thin film oven) aging conditions, using penetration, softening point, ductility, and mass loss parameters. In the asphalt binder mass loss criterion, it was determined by the percent weight loss due to loss of volatiles at high temperatures, which was measured based on the Eq. (3). Based on the superpave specifications [41], the recommended mass loss due to RTFO must be less than 1%.

$$\text{mass loss} = \frac{\text{weight before aging} - \text{weight after aging}}{\text{weight before aging}} \times 100\% \quad (3)$$

After testing the characteristics of glycerin pitch and pen 60/70 asphalt before and after adding GP based on the chemical structure and rheology of the asphalt, then testing the effect of using pen 60/70 asphalt substituted for GP on the stone matrix asphalt mixture was carried out. Where pen 60/70 asphalt + glycerin pitch is used as a binding agent for aggregate in the SMA mixture. The experimental procedures carried out in this study can be seen in Fig. 3.

3 Result and discussion

3.1 Glycerin pitch

Before testing the effect of GP on asphalt pen 60/70, it is necessary to test the characteristics of the glycerin pitch which can be seen in Table 1 [42]. An overview of the glycerin pitch can be seen in Fig. 4.

Based on the results of the glycerin pitch test in Table 1, it is known that the pH of GP is greater than 10, which means that GP has alkaline properties, the higher the pH, the greater the solubility of the catalyst salt in glycerin pitch which causes a high ash content [18]. The ash content is



Fig. 2 Hot plate magnetic stirrer device

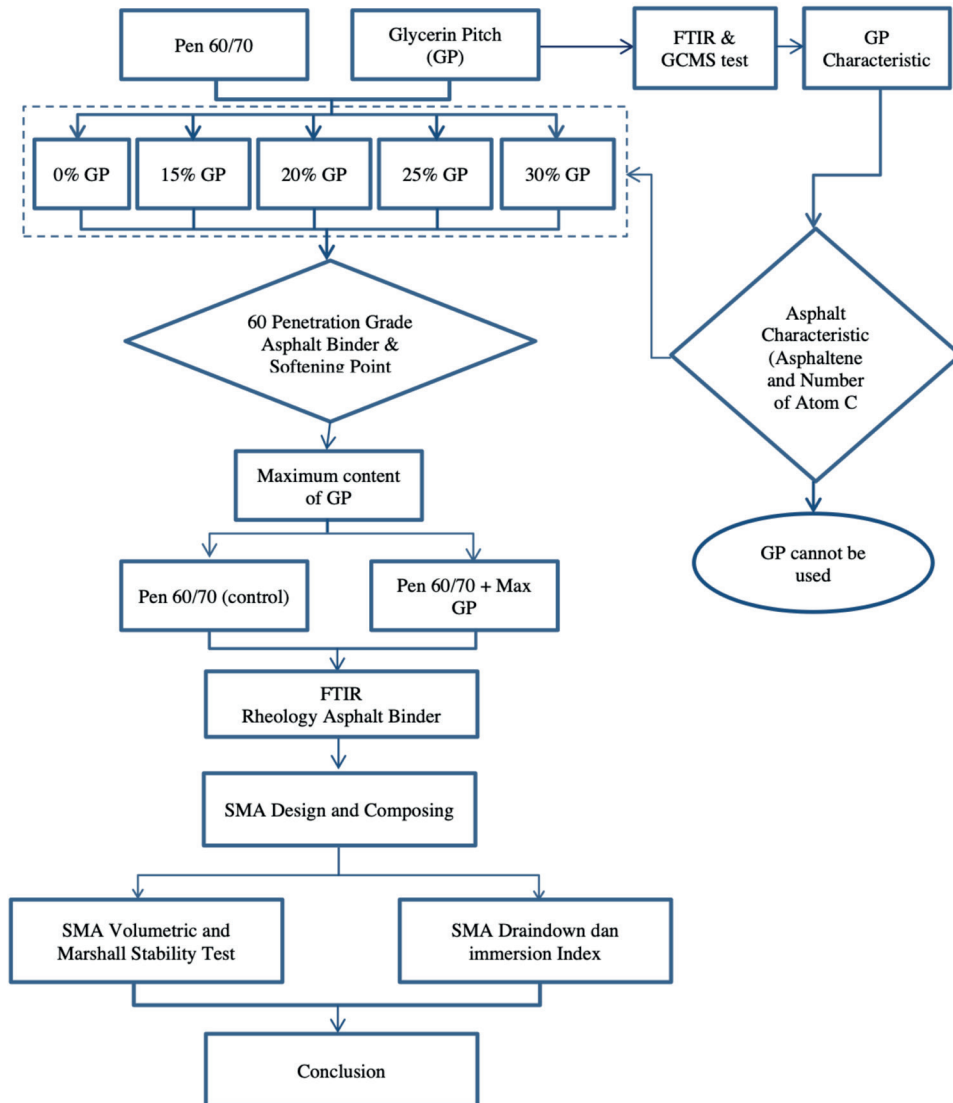


Fig. 3 Flowchart of the experimental procedure

Table 1 Characteristics of glycerin pitch

Test Parameter	Unit	BS 2622:1979 [42]	Result
PH	-	-	> 10 (Base)
Glycerol	%	> 80	13.02
Moisture content	%	No	2.56
Ash content	%	< 1.0	35
Saponification rate	-	-	-
Matter organic non glycerol (MONG)	%	< 1.5	14.45
Appearance	-	-	Brownish solid



Fig. 4 Glycerin pitch

35% indicating that the content of inorganic matter such as potassium salts probably comes from SW purification (HCl and NaOH). The GP saponification number indicates that the non-glycerol organic material does not have saponified compounds such as oil or fat, or perhaps because the

triglyceride value is too small. The organic content is 14.45%, the content of inorganic compounds in GP from the results of the X-Ray Diffraction test shows that the largest non-organic content is NaCl of 47.44%, the rest is 26.13% CaCO₃ compound and 26.43% NaO₂ compound.

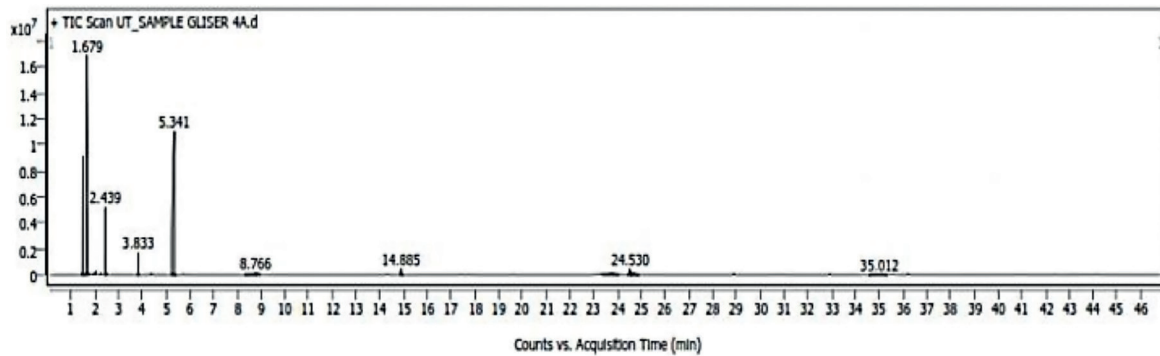


Fig. 5 Spectrum of GCMS glycerin pitch test results

In the GCMS test results, the number of C atoms is 24 (Fig. 5 and Table 2), which means that GP is included in bitumen materials such as the result of petroleum distillation, this is one of the factors that can be used as the basis that GP has the potential as asphalt substitute material.

3.2 Asphalt binder substitution penetration and softening point

The results of adding GP to asphalt pen 60/70 are seen based on the results of the penetration test and softening point, from the results of the penetration test (Fig. 6) it is known that higher the GP content added to asphalt pen 60/70 decreases the penetration value of asphalt, the maximum level of GP that produces asphalt with a penetration value that is still within the range of the pen 60/70 asphalt standard is 22%.

While the effect on the softening point of asphalt pen 60/70 (Fig. 7), it is known that higher the GP content added to pen 60/70 will increase the softening point value, at a GP level of 22%, the resulting softening point value is 52.2 °C which is still in accordance with the softening point specification standard for asphalt pen 60/70 which is ≥ 48 °C.

Based on penetration test and softening point of the asphalt, it is known that the maximum grade of GP as an asphalt pen 60/70 substitution is 22% by weight of asphalt. Furthermore, for testing the chemical structure and the other rheological parameters of asphalt will be tested at the 22% GP content.

3.3 Asphalt binder substitution chemical structure by FTIR

Asphalt chemical structure testing was carried out using FTIR on glycerin pitch, pen 60/70 asphalt and pen 60/70 asphalt +22% glycerin pitch. Then the test results were analyzed to identify the chemical structure of each type of asphalt compared to the chemical structure of pure asphaltene based on the results of the FTIR test by

Leon–Bermudes and Salazar [43]. The FTIR test results of the three types of asphalt compared to the pure asphaltene are shown in Fig. 8 and Table 3.

Table 2 Compound content in glycerin pitch

Compound	RM
Methyl alcohol	CH_4O
Acetone	$\text{C}_3\text{H}_6\text{O}$
2,2 – Dimethoxypropane	$\text{C}_5\text{H}_{12}\text{O}_2$
Glycerol	$\text{C}_3\text{H}_8\text{O}_3$
Diacetone alcohol	$\text{C}_6\text{H}_{12}\text{O}_2$
Diglycerol	$\text{C}_6\text{H}_{14}\text{O}_5$
Number of C Atoms	24

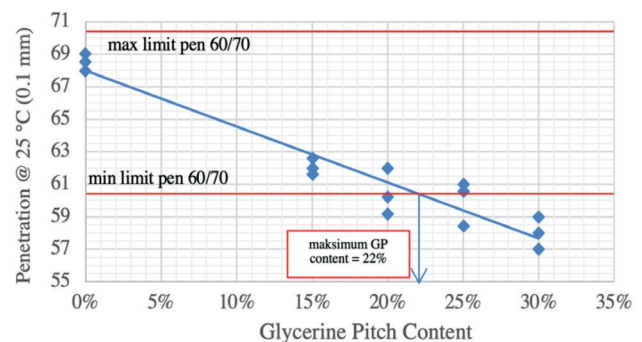


Fig. 6 Effect of adding GP on asphalt penetration pen 60/70

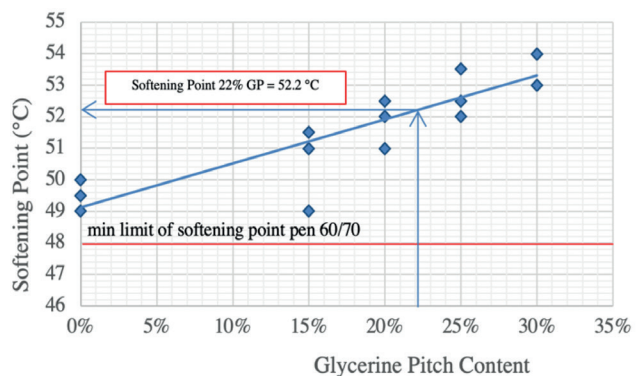


Fig. 7 Effect of addition of GP on softening point asphalt pen 60/70

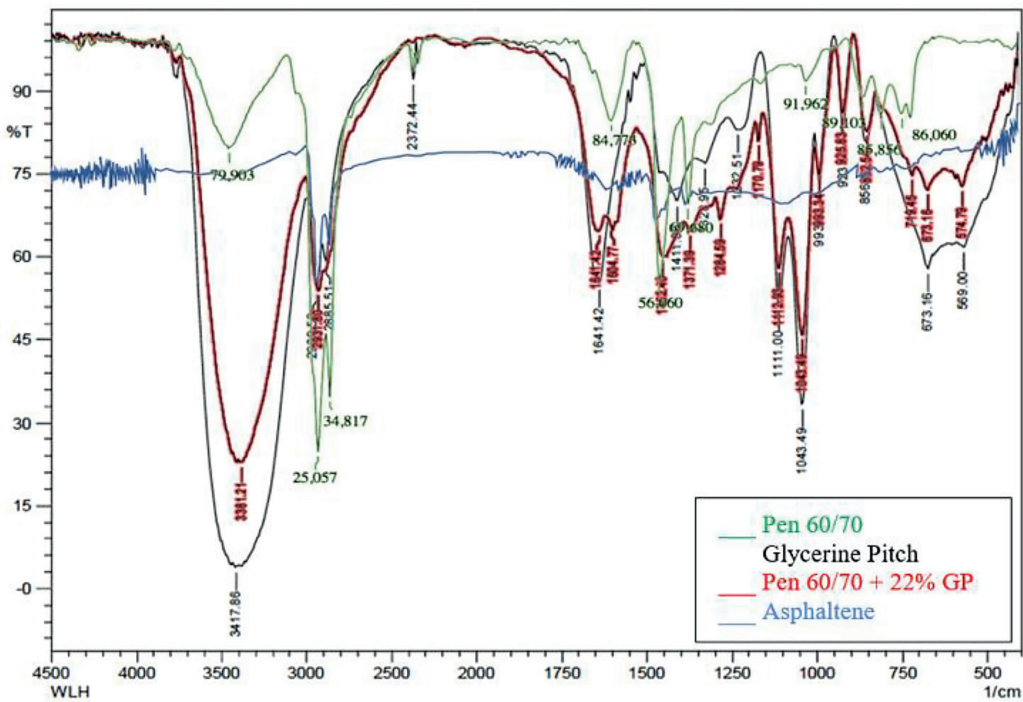


Fig. 8 All samples IR spectrum

Table 3 IR Spectrum all samples with asphaltene

Asphaltene IR spectrum (cm ⁻¹)	Samples		
	Pen 60/70	Glycerin Pitch	Pen 60/70 + 22% GP
3433	3448.72	3417.86	3381.21
2920	2922.16	2939.52	2931.80
1621	1600.79	1641.42	1641.42
1456	1460.11	-	1452.40
1373	1375.25	1328.95	1371.39
859	864.11	856.39	852.54

Based on the IR spectrum Table and Chart by Skoog et al. [44] and the compound functional group table by Coates in 2006 [45], known from Fig. 8 (green line), namely asphalt pen 60/70 showing -OH stretching groups which identify the presence of alcohol and phenol groups, the addition of 22% glycerin pitch increases the number of these groups. Other groups contained in asphalt pen 60/70 + 22% GP are C-H bending and C-H stretching which identify alkanes, besides that there is also an aromatic group = C-H₂. The carbonyl group is also present in the asphalt mixture and finally the C=C group which identifies the presence of ketones, alkenes, aldehydes, or carboxylic acids. The O-H stretching groups increase with each addition of glycerin pitch to the asphalt which increases the absorbance value, this indicates compounds such as acetone, carboxylic acids, esters, phenolic monomers evaporate during the analysis process so that more and more

aromatic groups are identified. This aromatic group is identical to most of the groups found in asphalt.

In Table 3, it is known that the IR spectrum of pure asphaltene is entirely in pen 60/70. The results show that pen 60/70 based on its chemical structure is asphalt, whereas in glycerin pitch, there is 1 (one) IR spectrum from pure asphaltene which it does not have, namely in the IR spectrum of 1456 cm⁻¹. However, after being added to pen 60/70, the entire IR spectrum of pure asphaltene is found in pen 60/70 + 22% GP, this shows that the addition of glycerin pitch to asphalt pen 60/70 does not change the chemical structure of asphalt so that GP can be used as an extender.

A part from being based on the IR spectrum of asphaltene, an analysis of changes in the chemical structure of asphalt pen 60/70 after adding GP was carried out by calculating the carbonyl index and sulfoxide index using Eqs. (2) and (3). Changes in the I_c and I_s values indicate that a modification of the asphalt occurs after adding added ingredients. in Table 4 and Fig. 9, it is known that the I_c and I_s values have increased when compared to before pen 60/70 and GP were mixed, this could indicate a change in chemical structure.

Table 4 Carbonyl index and sulfoxide index of asphalt binder

Asphalt Binder Types	Carbonyl Index (I _c)	Sulfoxide Index (I _s)
Pen 60/70	0.00461	0.01776
Glycerin Pitch (GP)	0.00880	0.01302
Pen 60/70 + 22%GP	0.01879	0.03905

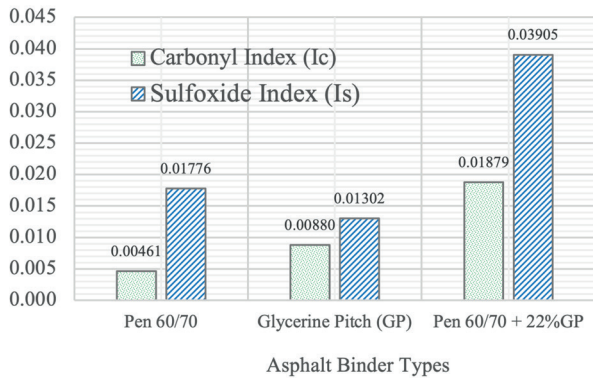


Fig. 9 Carbonyl index and sulfoxide index of asphalt binder

3.4 Physical and rheological properties of asphalt binder substitution

Apart from being tested based on its chemical structure, the addition of GP as an asphalt substitution for pen 60/70 at 22% content was also tested based on its rheological properties. The results of the asphalt rheology testing are shown in Table 5. Apart from the original conditions, the tests were also carried out under aged conditions or rolling thin film oven (RTFO) tests.

Based on the asphalt rheological test results shown in Table 5, it is known that the addition of GP up to 22% to the weight of pen 60/70 asphalt still produces asphalt that complies with the specifications of pen 60/70 asphalt (AASHTO M 20-70 [46] and ASTM D946 [47]). Fig. 10(a)–(c) illustrate the changes in the physical properties of pen 60/70 after adding glycerin pitch, however, in both original and aged conditions (RTFO), the changes that occur in the physical properties of pen 60/70 after adding glycerin pitch are still appropriate. with asphalt pen specifications of 60/70, so it can be seen that the addition of glycerin pitch up to 22% can still maintain the characteristics of asphalt in RTFO conditions. This shows that glycerin pitch based on the rheological test results of asphalt can be used as an asphalt substitution.

3.5 SMA mixtures

The use of GP in SMA asphalt mixture is as a substitute or extender for asphalt pen 60/70. Based on the Marga General Specification, 2018, the binder used must have pen 60/70 asphalt specifications for the SMA mixture. Based on the results of asphalt testing, it is known that the maximum GP content that can be substituted is 22%.

So that in the asphalt mixture are made into 3 types of variables of asphalt mixture according to the specifications of SMA, as follows:

1. SMA with pen 60/70 asphalt binder + 0% GP = SMA + 0% GP
2. SMA with asphalt binder pen 60/70 + 15% GP = SMA + 15% GP
3. SMA with asphalt binder pen 60/70 + 22% GP = SMA + 22% GP

The asphalt mixture is designed at P_b conditions (reference asphalt content, P_b) based on Eq. (4) according to the SMA mix design gradation (Table 6 and Fig. 11).

$$P_b = 0.035(\%CA) + 0.045(\%FA) + 1.18(\%Filler) + K, \quad (4)$$

where:

P_b (%) = reference asphalt content, percent by weight of the mix,

CA (%) = percent coarse aggregate (percentage of retained aggregate No. 4 (4.75 mm)),

FA (%) = percent of fine aggregate (percentage of aggregate passing No. 4 (4.75 mm) and retained on sieve No. 200 (0.075 mm)),

$Filler$ (%) = percent filler (percentage of aggregate passing No. 200 (0.075 mm)),

K = constant (0.5–1.0).

Table 5 Rheology properties of pen 60/70 and pen 60/70 + 22% GP

Test	Unit	Pen 60/70 Specification	Pen 60/70	Std Dev	Pen 60/70 + 22% GP	std Dev
Penetration, 25 °C, 100 gr, 5 sec	0.1 mm	60 - 70	68	0.764	60	0.764
Kinematic Viscosity 135 °C	cSt	> 300	512.6	8.053	459.96	5.341
Softening Point	°C	> 48	50	0.577	52.2	1.000
Ductility, 25 °C, 5 cm/minute	cm	> 100	> 100	-	> 100	-
Flash point with Clevelen Open Cup	°C	> 232	340	1.528	325	4.359
Solubility in Trichloroethylene	%	> 99	100.5	1.274	125	8.386
Specific gravity	-	> 1	1.04	0.018	1.061	0.002
Mass Loss	% Weight	< 0.8	0.149	0.008	0.24	0.081
Penetration RTFO	0.1 mm	> 54	58.8	0.361	55.5	1.500
Softening Point RTFO	°C	-	56	1.000	57	0.961
Ductility RTFO	Cm	> 100	>100	0.577	> 100	1.528

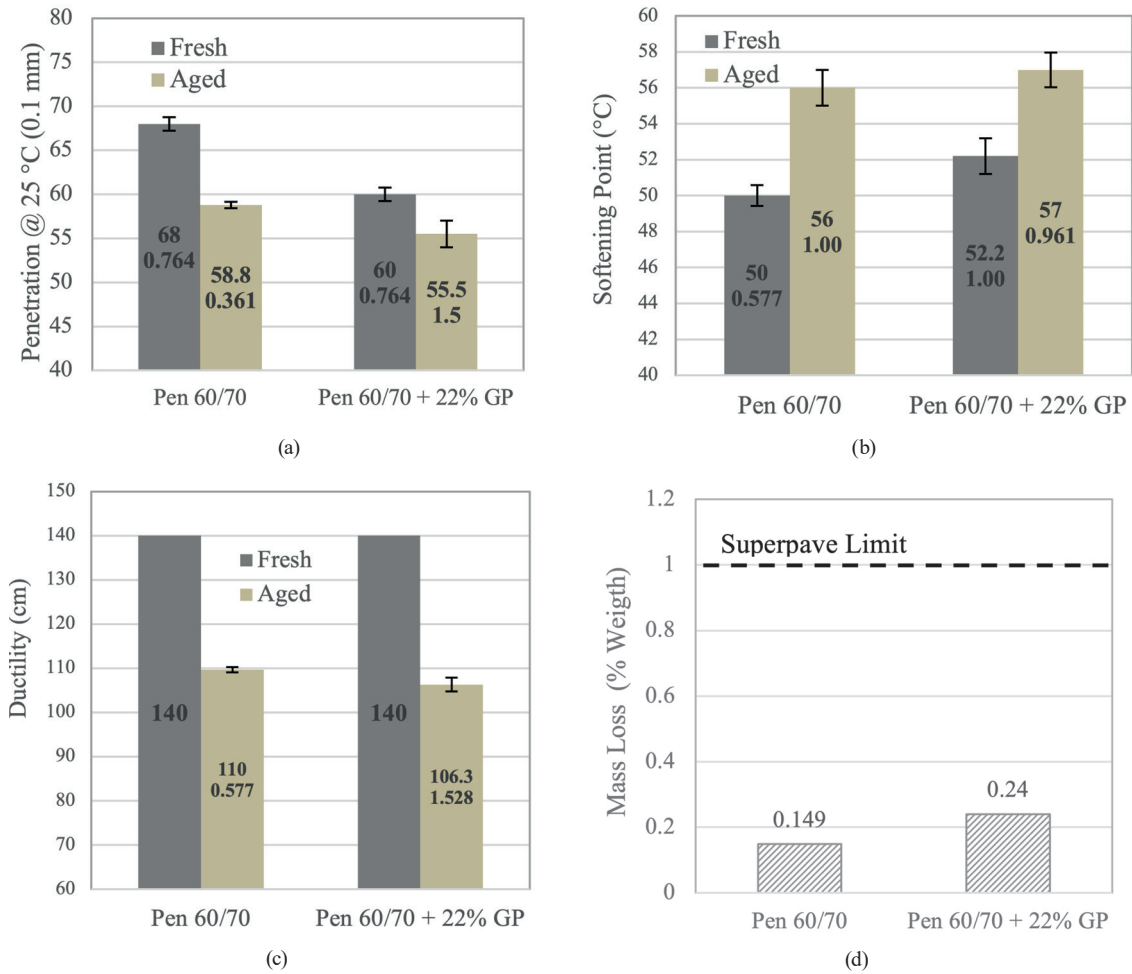


Fig. 10 Physical properties of asphalt binder before and after RTFO, (a) penetration, (b) softening point, (c) ductility, (d) mass loss

Based on the gradation of the SMA mix aggregate design, the P_b value is 7%, then the mixture is made using this asphalt content. Tests carried out on the SMA mixture in this study consisted of volumetric, stability, flow, immersion index, and draindown tests in accordance with the Indonesian Highway General Specification, 2018 [22]. The number of test objects made is described in Table 7. The material used to make Marshall test objects is

Table 6 SMA Gradation design mixtures

Sieve Number	SMA Specification	SMA Gradation (%)	SMA Gradation Design
11/2	25	100	100.0
3/4"	19	90-100	95.0
1/2"	12.5	50-88	69.0
3/8"	9.52	25-60	42.5
No. 4	4.76	20-28	24.0
No. 8	2.36	16-24	20.0
No. 200	0.075	8-11	9.5

- Pen 60/70 is asphalt from the distillation of petroleum which is used as the main binder in the SMA mixture, according to the Indonesian Highway General Specifications 2018.
- Glycerin pitch is an oleochemical waste as a substitute for binder for SMA mix asphalt, the result of the substitution is in accordance with the specifications for SMA mix binder according to the Bina Marga General Specifications, 2018.

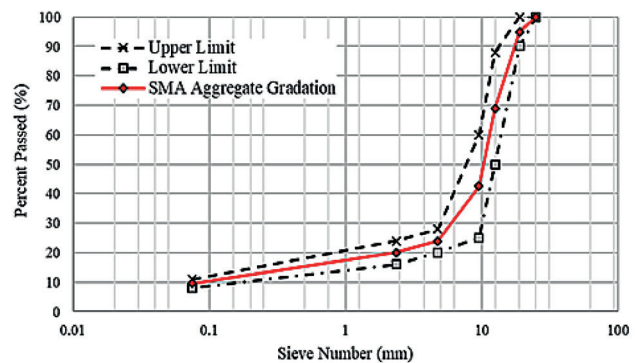


Fig. 11 Aggregate Gradation of Stone Mastic Asphalt

- c. Viatop66 cellulose fiber is the main carbohydrate synthesized by plants and occupies almost 60% of the components of the wood structure, as an added material to assist the granulation process and as a filler in the space between fibers needed to complete the dispersion process on the fibers during the asphalt mixing process. The specification requirements for cellulose fiber are in accordance with SNI 8129:2015.
- d. Aggregates from Batujajar, West Java Province, Indonesia whose sizes are adjusted to the gradation of the SMA mix plan according to the Bina Marga General Specifications, 2018.

The SMA mixture was prepared by first preparing asphalt as a binder which was made from pen 60/70 asphalt with 3 variations of GP substitution namely 0%, 15%, and 22%. The use of variations in the amount of GP was carried out to see the effect of using GP as a substitute for asphalt on the characteristics of the SMA mix. Furthermore, the asphalt is heated according to the mixing viscosity (Table 8), the aggregate is heated at a mixing temperature of 28 °C, then the aggregate and asphalt along with the cellulose fiber are mixed at the mixing temperature for 30–60 seconds until it reaches the compaction temperature, put it into the Marshall mold, then compacted with the number of blows on each side as much as 50 times. Furthermore, the sample is removed from the mold and can

be tested volumetrically, before being tested for Marshall stability, the test object is coated with paraffin and then immersed in a water bath at a maintained temperature of 60 ± 1 °C for 30 minutes following the Marshall stability test procedure according to SNI 8129: 2015 [48].

In the immersion index test, the specimens were made the same as the Marshall test specimens, the difference was the immersion time when the Marshall stability was to be tested, in this study, the immersion time was carried out for 30 minutes and 24 hours.

For the draindown test, asphalt, aggregate, and cellulose fiber pellets are mixed at mixing temperature without compaction, then put into a wire basket with an aluminum base (Fig. 12), standard basket made according to AASHTO T305 [49], which is 165 ± 16.5 mm deep, 108 ± 10.8 mm wide, with basket bottom 25 ± 2.5 mm from the bottom of the wire basket assembly, constructed using standard 0.25 inch (6.3 mm) sieve cloth as specified in AASHTO M 231 [50]. Store the test object in the oven at $120\text{--}175$ °C ± 2 °C for 1 hour ± 5 minutes. If the specimen has cooled to more than 25 °C before being put into the oven, it only needs to be in the oven for 70 ± 5 minutes. After that, remove the specimen from the oven and separate the wire basket and aluminum mat then weigh the weight of the aluminum mat which has been filled with melted asphalt mixture. Then a calculation analysis is carried out to determine the percentage of draindown values.

The National Asphalt Pavement Association (NAPA), states draindown is a condition where aggregate and asphalt separate from the sample as a whole and flow down from the mixture (asphalt drainage) [51]. The draindown test is more significant for dense and conventionally graded SMA mixtures. This draindown test is used to determine the amount of asphalt mixture draindown according to predetermined specifications. The draindown test was developed by AASHTO T305 [49], which is a test to anticipate conditions that may occur during the production, storage, transportation and spread of asphalt mixtures.

Table 7 Type of test and number of samples

Mixture Types	Type of Tests	Samples
SMA + 0%GP	Volumetric and Marshall's Stability	3
	Immersion Index	6
	Draindown	3
SMA + 15%GP	Volumetric and Marshall's Stability	3
	Immersion Index	6
	Draindown	3
SMA + 22%GP	Volumetric and Marshall's Stability	3
	Immersion Index	6
	Draindown	3
Total	36	

Table 8 The mixing temperature of the SMA mixture is based on the results of the asphalt viscosity test

Mixture Types	Mixing Temperature (°C)	Compaction Temperature (°C)
SMA + 0%GP	154.5	144.5
SMA + 15%GP	158.0	144.0
SMA + 22%GP	168.0	151.5

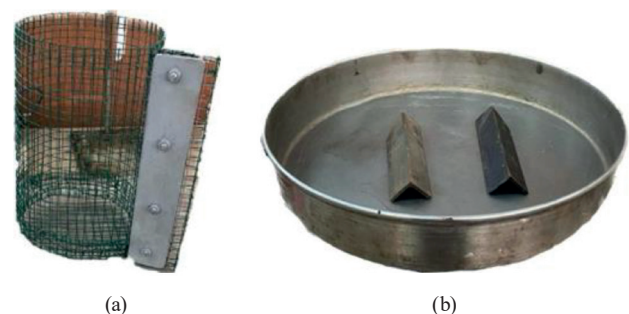


Fig. 12 Draindown test apparatus; (a) Draindown basket, (b) Buffer plat

The results of testing the SMA mixture for each GP content are described in Fig. 13 and Table 9. These values are the average of each test object made for each type of mixture.

Based on the test results of the SMA mix using glycerin pitch as a substitute for asphalt pen 60/70 with various variations, it can be seen that:

1. Based on the density value, the addition of GP levels indicates a change in the density value
2. Based on volumetric asphalt mix, it is known that based on VMA and VIM, the value increases after the GP percentage is 15% and decreases when the GP is 22%, this condition is inversely proportional to VFB.

3. The stability of the SMA mixture for each mixture is included in the specification, which is greater than 600 Kg, the stability value shows an increase in line with the increase in the GP content in the asphalt. However, the flow of each mix is not included in the SMA mix specifications.
4. The immersion index of the SMA mixture using GP as a substitute for asphalt pen 60/70, shows an increase at 15% GP and decreased at 22% GP. Based on the immersion index, the mixture of SMA + 15% GP is a mixture with an immersion index according to specifications.

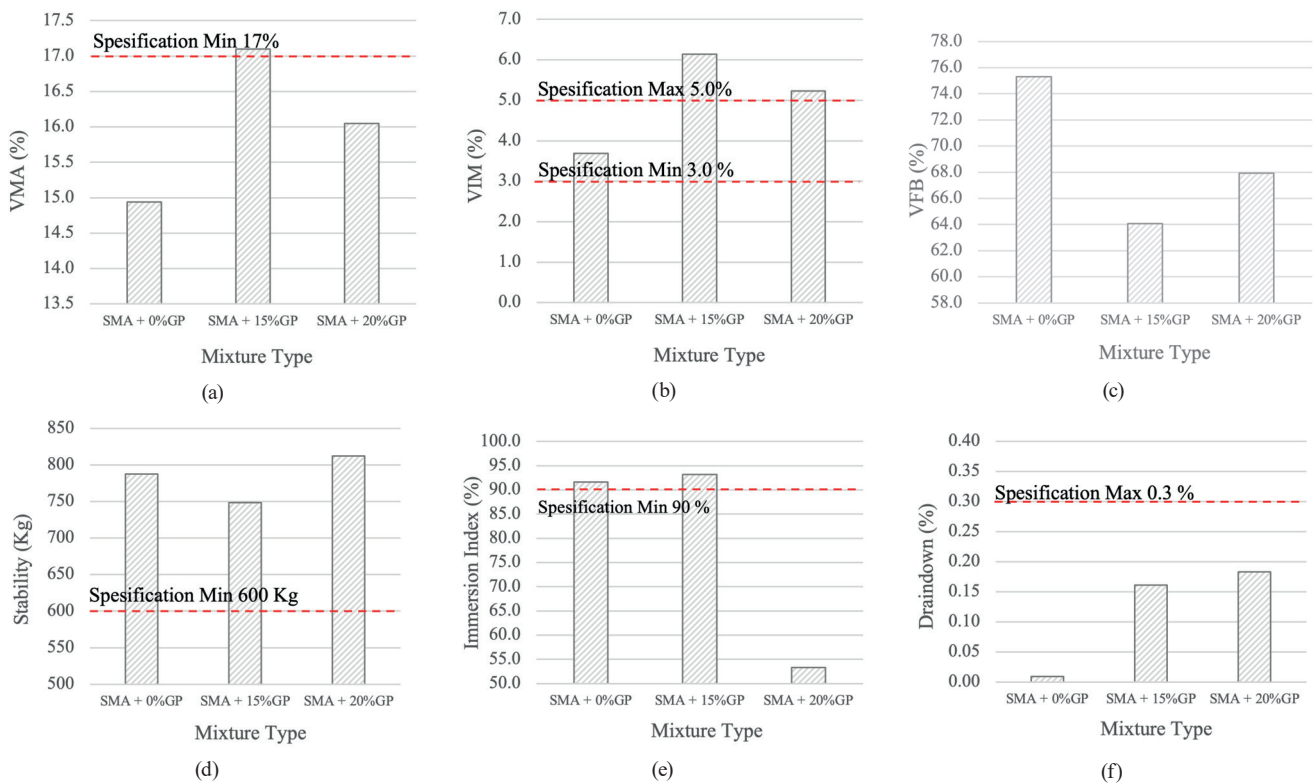


Fig. 13 SMA mixture characteristics based on glycerin pitch content; (a) Void in mineral aggregate, (b) Void in mix, (c) Void filled with bitumen, (d) Stability, (e) Marshall immersion index, (f) Drainddown

Table 9 Characteristics of the SMA mixture with variations of glycerin pitch content

Mixture Characteristics	Units	Specification [22]	Test Results		
			SMA + 0%GP	SMA + 15%GP	SMA + 20%GP
Asphalt Content	%	-	7	7	7
Density	gr/cc	-	2.25	2.19	2.22
Void in Mineral Aggregate, VMA	%	min 17	14.94	17.10	16.05
Void In Mixture, VIM	%	3.0–5.0	3.69	6.14	5.23
Void Filled with Bitumen, VFB	%	-	75.30	64.08	67.93
Stability	Kg	600	787.5	748.3	812.5
Flow	mm	2.0–4.5	8.8	6.92	5.66
Immersion Index	%	> 90	91.61	93.20	53.31
Draindown	%	< 0.3	0.009	0.161	0.183

In draindown, it is known that based on the specifications, the SMA mix has a maximum draindown value of 0.3%. The test results show that all types of mixtures have draindown according to specifications, an increase in GP content on asphalt pen 60/70 affects SMA mixtures with an increased draindown value.

Overall, from the results of testing the SMA mix using glycerin pitch substituted asphalt, it is the SMA mix with 15% GP that has the characteristics best suited to the SMA asphalt mix specifications according to the Indonesian Highway General Specification, 2018 [22].

Although in terms of the chemical structure and rheology of asphalt, the addition of GP up to 22% produces asphalt that meets the specifications of pen 60/70 asphalt, but in its application in SMA asphalt mixtures, the most appropriate GP content to use is 15% by weight of pen 60/70 asphalt binder.

3.6 Tukey–Kramer statistical grouping

Tukey–Kramer statistical grouping analysis [52, 53], was carried out to see and compare the significance of the effect of glycerin pitch on each mixture based on its characteristics. Significance was analyzed for the characteristics of the SMA mix, namely stability, immersion index, and draindown. Tables 10–12 show the results of the Tukey–Kramer Statistical Grouping analysis for SMA mixtures using glycerin pitch as a substitute for asphalt pen 60/70.

Based on Table 10, the significance of using GP as an extender/substitute for asphalt binder pen 60/70 on the stability of the asphalt mixture does not have a significant effect on the entire mixture, but when compared to the three, the use of GP has a significant effect on SMA + 22% GP mixture, this can be seen from the absolute value the mean different stability of the mixture is greater than that of other SMA mixtures. While on the characteristics of the

Table 10 Results of the Tukey–Kramer statistical grouping analysis of stability of SMA + GP mixtures

Mixture Type	Count	Sum	Average	Variance
SMA + 0% GP	3	2362.49	787.49	26758.95
SMA + 15% GP	3	2244.81	748.27	16645.49
SMA + 22% GP	3	2426.41	808.80	164.65
Comparison	Abs. Mean Diff	Q critical value	Significant	
SMA + 0%GP vs SMA + 15% GP	39.22	128.55	no	
SMA + 15%GP vs SMA + 22% GP	60.53	128.55	no	
SMA + 0%GP vs SMA + 22% GP	21.31	128.55	no	

Table 11 Results of the Tukey–Kramer statistical grouping analysis of immersion index of SMA + GP mixtures

Mixture Type	Count	Sum	Average	Variance
SMA + 0% GP	3	279.96	93.32	152.79
SMA + 15% GP	3	289.67	96.56	1160.21
SMA + 22% GP	3	158.42	52.81	222.63
Comparison	Abs. Mean Diff	Q critical value	Significant	
SMA + 0% GP vs SMA + 15% GP	3.24	24.13	no	
SMA + 15% GP vs SMA + 22% GP	43.75	24.13	yes	
SMA + 0% GP vs SMA + 22% GP	40.51	24.13	yes	

Table 12 Results of the Tukey–Kramer statistical grouping analysis of draindown of SMA + GP mixtures

Mixture Type	Count	Sum	Average	Variance
SMA + 0% GP	3	0.0261	0.0087	0.0001
SMA + 15% GP	3	0.4817	0.1606	0.0328
SMA + 22% GP	3	0.5484	0.1828	0.0018
Comparison	Abs. Mean Diff	Q critical value	Significant	
SMA + 0% GP vs SMA + 15% GP	0.15	0.11	yes	
SMA + 15% GP vs SMA + 22% GP	0.02	0.11	no	
SMA + 0% GP vs SMA + 22% GP	0.17	0.11	yes	

immersion index (Table 11), the use of GP has a significant effect on the mixture of SMA + 0% GP and SMA + 15% GP against SMA + 22% GP. Furthermore, in the analysis of the significance of GP on the draindown characteristics of the SMA mixture (Table 12), it is known that GP has a significant effect on the draindown of the SMA + 0% GP mixture.

4 Conclusions

Based on the test results of GP as asphalt substitute it can be concluded, as follows:

1. The characteristics of GP based on chemical structure and atomic content of C showed that GP has an IR spectrum and atomic content of 24 C, this indicates that GP has the potential like asphalt binder.
2. Based on the results of the penetration test and softening point, the maximum content of GP as an asphalt substitution is 22% by weight of pen 60/70 asphalt.

References

- [1] Anusha, T. M., Avaradi, S., Jagadeesh, H. S. "Effective Utilization of Reclaimed Asphalt Pavement by using Rejuvenator in Stone Mastic Asphalt For Binder Course", 10(9), pp. 458–463, 2021. <https://doi.org/10.17577/IJERTV10IS090095>
- [2] Kumar, S., Prasad, N. S. "Experimental Investigation on Stone Matrix Asphalt Mixture using Arbocel Fiber", International Journal of Science and Research (IJSR), 6(9), pp. 885–892, 2015.
- [3] EAPA "Heavy Duty Surfaces the Arguments for SMA", European Asphalt Pavement Association, Brussels, Belgium, 2018.
- [4] Al-Hadidy, A., Tan, Y. "Performance of the SMA mixes made with the various binders", Construction and Building Materials, 25(9), pp. 3668–3673, 2011. <https://doi.org/10.1016/j.conbuildmat.2011.03.008>
- [5] Hainin, R., Reshi, W. F., Niroumand, H. "The importance of stone mastic asphalt in construction", Electronic Journal of Geotechnical Engineering, 17, pp. 49–56, 2012.
- [6] Kiran Kumar, N. L. N., Ravitheja, A. "Characteristics of stone matrix asphalt by using natural fibers as additives", Materials Today: Proceedings, 19(2), pp. 397–402, 2019. <https://doi.org/10.1016/j.matpr.2019.07.624>
- [7] Sarang, G., Lekha, B. M., Geethu, J. S., Ravi Shankar, A. U. "Laboratory performance of stone matrix asphalt mixtures with two aggregate gradations", Journal of Modern Transportation, 23(2), pp. 130–136, 2015. <https://doi.org/10.1007/s40534-015-0071-5>
- [8] Ridwan, M. "Kementerian PUPR: 83 Persen Kebutuhan Aspal RI Masih Impor", (PUPR Ministry: 83 Percent of RI's Asphalt Needs are Still Imported), 2022. [online] Available at: <https://ekonomi.bisnis.com/read/20221011/45/1586512/kementerian-pupr-83-persen-kebutuhan-aspal-ri-masih-impor> (in Indonesian)
- [9] Pérez, I., Pasandín, A. R., Pais, J. C., Pereira, P. A. A. "Feasibility of Using a Lignin-Containing Waste in Asphalt Binders", Waste and Biomass Valorization, 11, pp. 3021–3034, 2020. <https://doi.org/10.1007/s12649-019-00590-4>
- [10] Harman, T., Youtcheff, J., Bukowski, J. "An Alternative Asphalt Binder, Sulfur-Extended Asphalt (SEA)", U.S. Department of Transportation, Federal Highway Administration, Washington, DC, USA, FHWA-HIF-12-037, 2012.
- [11] Wu, J., Liu, Q., Wang, C., Wu, W., Han, W. "Investigation of lignin as an alternative extender of bitumen for asphalt pavements", Journal of Cleaner Production, 283, 124663, 2021. <https://doi.org/10.1016/j.jclepro.2020.124663>
- [12] Pérez, I. P., Rodríguez Pasandín, A. M., Pais, J. C., Alves Pereira, P. A. "Use of lignin biopolymer from industrial waste as bitumen extender for asphalt mixtures", Journal of Cleaner Production, 220, pp. 87–98, 2019. <https://doi.org/10.1016/j.jclepro.2019.02.082>
- [13] Teoh, W. P., Chee, S. Y., Habib, N. Z. Bashir, M. J. K., Chok, V. S., Ng, C. A. "Chemical investigation and process optimization of glycerine pitch in the green production of roofing tiles", Journal of Building Engineering, 43, 102869, 2021. <https://doi.org/10.1016/j.jobe.2021.102869>
- [14] Soerawidjaja, T. H. "Spectrum of New Uses for Glycerol", [pdf] Jakarta, Indonesia, 2019. Available: <https://sawitindonesia.com/wp-content/uploads/2019/11/pembicara-2-Dr-Ir-Tatang-Hernas-S.pdf> (in Indonesian)
- [15] Hidawati, E. N., Sakinah, M. M. "Treatment of Glycerin Pitch from Biodiesel Production", International Journal of Chemical and Environmental Engineering, 2(5), pp. 309–313, 2011.
- [3] In terms of asphalt rheology, it is known that the addition of GP as asphalt binder extender/substitutions still provides asphalt binder characteristics that comply with the standard specifications for asphalt binder pen 60/70.
- [4] The most appropriate glycerin pitch content to be used as a substitute for asphalt pen 60/70 to produce SMA asphalt mixture according to specifications is 15% by weight of asphalt.
- [5] By Tukey-Kramer analysis, in stability characteristics of SMA, GP has a significant effect on SMA + 22% GP mixture. In the immersion index, the use of GP has a significant effect on all type of mixture, while in the draindown characteristics has significant effect on the SMA + 0% GP mixture.

Acknowledgement

This research is supported by Center of Research and Community Services, Bandung State Polytechnic, Indonesia.

- [16] Sipayung, T., Purba, J. H. V. "Palm Oil Agribusiness Economics", Palm Oil Agribusiness Strategic Policy Institute, 2015. [online] Available at: <https://palmoilina.asia/pustaka-digital/ekonomi-agribisnis-minyak-sawit/> (in Indonesian)
- [17] APOLIN (Association of Indonesian Oleochemical Producers) "Apolin: The capacity of Indonesia's oleochemical industry is the largest in the world", Antara News, 2021. (in Indonesian)
- [18] Hazimah, A. H., Ooi, T. L., Salmiah, A. "Recovery of Glycerol and Diglycerol from Glycerol Pitch", *Journal of Oil Palm Research*, 15(1), pp. 1–5, 2003.
- [19] ASTM "ASTM C1942-03 Standard Specification for Concrete Roof Tile", ASTM International, West Conshohocken, PA, USA, 2009.
- [20] ASTM "ASTM C1167-03 Standard Specification for Standard Specification for Clay Roof Tiles Clay Roof Tiles", ASTM International, West Conshohocken, PA, USA, 2003.
- [21] Tong Jian, B. Y. "Utilization of Glycerine Pitch as a Binder in the Production of Green Roofing Tile", BSc Thesis, Tunku Abdul Rahman University, 2019.
- [22] Directorate General of Highways "General Specification of Bina Marga 2018 For Road and Bridge Construction Work (Revision 2)", Directorate General of Highways, Ministry of Public Work and Housing, Jakarta, Indonesia, 2018. (in Indonesian)
- [23] ASTM "ASTM C494-03 Standard Specification for Chemical Admixtures for Concrete", ASTM International, West Conshohocken, PA, USA, 2013.
- [24] ASTM "ASTM D2974-14 Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils", ASTM International, West Conshohocken, PA, USA, 2014. <https://doi.org/10.1520/D2974-14>
- [25] Hill Laboratories "Hydrocarbon Analysis for Environmental Samples", Hamilton, New Zealand, 2241v4, 2022.
- [26] Kasick, A. "Gas Chromatography-Mass Spectrometry Study of a Painting That May Contain Asphaltum Pigment", Research Honors Program Thesis, Marietta College, 2013.
- [27] Moore, J. W., Stanitski, C. L., Jurs, P. C. "Chemistry: The Molecular Science", 2nd ed., Thompson, 2008. ISBN 13: 9780495112556
- [28] Tang, S. "Asphalt modification by utilizing bio-oil ESP and tall oil additive", MSc Thesis, Iowa State University, 2010.
- [29] Dizhbite, T., Telysheva, G., Jurkane, V., Viesturs, U. "Characterization of the radical scavenging activity of lignins - Natural antioxidants", *Bioresource Technology*, 95(3), pp. 309–317, 2004. <https://doi.org/10.1016/j.biortech.2004.02.024>
- [30] Appels, F. V. W., Camere, S., Montali, M., Karana, E., Jansen, K. M. B., Dijksterhuis, J., Krijgsheld, P., Wösten, H. A. B. "Fabrication factors influencing mechanical, moisture- and water-related properties of mycelium-based composites", *Materials & Design*, 161, pp. 64–71, 2019. <https://doi.org/10.1016/j.matdes.2018.11.027>
- [31] Yaro, N. S. A., Bin Napih, M., Sutanto, M. H., Usman, A., Saeed, S. M. "Performance evaluation of waste palm oil fiber reinforced stone matrix asphalt mixtures using traditional and sequential mixing processes", *Case Studies in Construction Materials*, 15, e00783, 2021. <https://doi.org/10.1016/j.cscm.2021.e00783>
- [32] Read, J., Whiteoak, D. "The Shell Bitumen Handbook", Thomas Telford, 2003. ISBN: 0-7277-3220-X <https://doi.org/10.1680/sbh.32200>
- [33] ASTM "ASTM D36/D36M-14 Standard Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus)", ASTM International, West Conshohocken, PA, USA, 2014. https://doi.org/10.1520/D0036_D0036M-14
- [34] ASTM "ASTM D5-05 Standard Test Method for Penetration of Bituminous Materials", ASTM International, West Conshohocken, PA, USA, 2005. <https://doi.org/10.1520/D0005-05>
- [35] ASTM "ASTM E1252 Standard Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis", ASTM International, West Conshohocken, PA, USA, 1998.
- [36] Sihombing, A. V. R., Subagio, B. S., Hariyadi, E. S., Yamin, A. "Chemical, morphological, and high temperature rheological behaviour of Bioasbuton® as an alternative binder for asphalt concrete in Indonesia", *Journal of King Saud University - Engineering Sciences*, 33(5), pp. 308–317, 2021. <https://doi.org/10.1016/j.jksues.2020.07.006>
- [37] Mills-Beale, J., You, Z., Fini, E., Zada, B., Lee, C. H., Yap, Y. K. "Aging influence on rheology properties of petroleum-based asphalt modified with biobinder", *Journal of Materials in Civil Engineering*, 26(2), pp. 358–366, 2014. [https://doi.org/10.1061/\(ASCE\)MT.1943-5533.0000712](https://doi.org/10.1061/(ASCE)MT.1943-5533.0000712)
- [38] Hofko, B., Porot, L., Cannone, A. F., Poulidakos, L., Huber, L., Lu, X., Mollenhauer, K., Grothe, H. "FTIR spectral analysis of bituminous binders: reproducibility and impact of ageing temperature", *Materials and Structures*, 51, 45, 2018. <https://doi.org/10.1617/s11527-018-1170-7>
- [39] Lugo-Lugo, V., Barrera-Díaz, C., Ureña-Núñez, F., Bilyeu, B., Linares-Hernández, I. "Biosorption of Cr(III) and Fe(III) in single and binary systems onto pretreated orange peel", *Journal of Environmental Management*, 112, pp. 120–127, 2012. <https://doi.org/10.1016/j.jenvman.2012.07.009>
- [40] Lamontagne, J., Dumas, P., Mouillet, V., Kister, J. "Comparison by Fourier transform infrared (FTIR) spectroscopy of different ageing techniques: Application to road bitumens", *Fuel*, 80(4), pp. 483–488, 2001. [https://doi.org/10.1016/S0016-2361\(00\)00121-6](https://doi.org/10.1016/S0016-2361(00)00121-6)
- [41] D'Angelo, J., Kluttz, R., Dongre, R. N., Stephens, K., Zanzotto, L. "Revision of the Superpave high temperature binder specification: The multiple stress creep recovery test", In: 2007 Journal of the Association of Asphalt Paving Technologists: From the Proceedings of the Technical Sessions, San Antonio TX, USA, 2007, pp. 123–162. [online] Available: <https://trid.trb.org/view/839413>
- [42] British Standard "BS 2622:1979 The British Standard Specification of Hydrolysis crude glycerol", British Standards Institution, 1979.
- [43] León-Bermúdez, A.-Y., Salazar, R. "Synthesis and characterization of the polystyrene – Asphaltene graft copolymer by FT-IR spectroscopy", *CT&F – Ciencia, Tecnología y Futuro*, 3(4), pp. 157–167, 2008.
- [44] Skoog, D. A., Holler, F. J., Crouch, S. R. "Principles of Instrumental Analysis", *Analytica Chimica Acta*, 152, 1983. [https://doi.org/10.1016/s0003-2670\(00\)84936-3](https://doi.org/10.1016/s0003-2670(00)84936-3)

- [45] Coates, J., "Interpretation of Infrared Spectra, A Practical Approach", Encyclopedia of Analytical Chemistry, John Wiley & Sons, 2006. ISBN: 9780471976707
<https://doi.org/10.1002/9780470027318.a5606>
- [46] AASHTO "AASHTO M 20-70 Standard Specification for Penetration-Graded Asphalt Cement", American Association of State and Highway Transportation Officials, 2004.
- [47] ASTM "ASTM D946, Standard Specification for Penetration-Graded Asphalt Cement for Use in Pavement", ASTM International, West Conshohocken, PA, USA, 1999.
<https://doi.org/10.1520/D0946>
- [48] "SNI 8129:2015 Specification for Stone Matrix Asphalt (SMA)", Indonesian National Standards, 2015. (in Indonesian)
- [49] AASHTO "AASHTO T 305-14 Standard Method of Test for Determination of Draindown Characteristics in Uncompacted Asphalt Mixtures", American Association of State Highway and Transportation Officials, Washington, DC, USA, 2000.
- [50] AASHTO "AASHTO M 231: 1995 (R2019) Standard Specification for Weighing Devices Used in the Testing of Materials", American Association of State Highway and Transportation Officials, Washington, DC, USA, 2019.
- [51] Roberts, F. L., Kandhal, P. S., Brown, E. R., Lee, D.-Y., Kennedy, T. W. "Hot Mix Asphalt Materials, Mixture Design, and Construction", 2nd ed., NAPA Education Foundation, 1996. ISBN-10: 0914313010
- [52] Tukey, J. W. "Comparing Individual Means in the Analysis of Variance", Biometrics, 5(2), pp. 99-114, 1949.
<https://doi.org/10.2307/3001913>
- [53] Kramer, C. Y. "Extension of Multiple Range Tests to Group Means with Unequal Numbers of Replications", Biometrics, 12(3), pp. 307-310, 1956.
<https://doi.org/10.2307/3001469>