GLYCERINE PITCH AS AN EXTENDER FOR ASPHALT BINDER PEN 60/70

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ABSTRACT

Glycerine pitch (GP) is a by-product from the refining process of Crude Glycerol from the palm oleochemical industry which in its processing requires a cost of USD 400/ton. In Indonesia, it is estimated that the production of GP reaches 35 thousand tons/year. This study aims to examine the potential of GP as an extender for asphalt binder pen 60/70, according to the Fourier transform infrared spectroscopy (FTIR) test to see its chemical structure and asphalt binder rheology in the laboratory. The materials used in this research are pen 60/70 and GP from the oil palm oleochemical industry in Bekasi Regency which are produced from the hydrolysis route. GP was added to asphalt pen 60/70 with variations in the percentage of GP to the weight of asphalt pen 60/70 were 0%, 15%, 20%, and 25%. Based on the results of the FTIR test, it is known that GP belongs to a polyglycerol compound which is similar to the long compound in petroleum asphalt. The addition of GP up to 25% to asphalt-based rheology still meets the characteristics of pen 60/70 with a penetration value of 64.14 dmm, softening point 52 □C, viscosity 408.52 cSt, ductility > 100 cm and density 1.061.

Keywords : Glycherine Pitch; Ashpalt; FITR

1. INTRODUCTION

The oleochemical industry has existed in Indonesia since 1975, during its production period produced oleochemical waste so it required an oleochemical waste treatment process, but until 2014 there were only 9 waste treatment plants with a processing capacity of 1.40 million tons/year (Rofiqi *et al*, 2018) (Sipayung and Purba, 2015). Palm oil-based oleochemical capacity is currently the largest in the world, reaching up to 23.3 million tons/year (Apolin, 2021). Oleochemicals are fatty oil derivative compounds obtained through a chemical process (Badan Pengelola Dana Perkebunan Sawit, 2018).
Along with the increase in the human standard of living, it is estimated that the demand for this oleochemical will increase significantly. Glycerine or glycerol is one of the oleochemical products whose use is widespread in industries such as pharmaceutical, medical, food and so on (W. P. Teoh *et al,* 2021). Every year Indonesia produces glycerol as much as 650,000 tons/year (Soerawidjaja, 2019). This glycerol purification process will produce a waste called glycerine pitch of as much as 3% (Hidawati and Sakinah, 2011).
Glycerine pitch (GP) is a dark brown viscous liquid containing non-glycerol organic compounds and has a high mineral content (Hazimah *et al,* 2003). Taking into account

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the possibility of an increase in glycerin production, which will result in the number of funds needed to process glycerin pitch will be even greater, a solution is needed to overcome this (A. Choirun Az Zahra *et al,* 2021). Several researchers have conducted research to increase the use value of GP so that it can be reused or find pathways for chemical glycerol purification processes to reduce the resulting waste (A. Choirun Az Zahra *et al,* 2021).

Physically, GP is brownish black with a gel-like viscosity similar to asphalt/tar/bitumen. Previous studies have used GP as a binder in producing roof tiles, with the potential as a binding material to produce tiles that meet the minimum requirements for tile strength based on ASTM C 1492-03 and water absorption according to ASTM C 1167-03, from the results of the research obtained dry values. tile strength of 5385.40 N and wet strength of 1139.96 N, and absorption with a value of 4.66% (B. Y. Tong Jian, 2019). In some countries, in the manufacture of roof tiles, many use asphalt as a binding material which is commonly called asphalt roof shingles, with the physical characteristics of GP being similar to asphalt, which is the reason for using GP as a binder in the manufacture of roof tiles. The use of GP as a binder for the manufacture of building materials is the basis of this study to utilize GP as a binder (asphalt) in asphalt mixtures, as the initial stage of using GP, this study aims to determine the potential of GP as an asphalt extender based on the properties of asphalt pen 60/70 which is the type of asphalt commonly used in Indonesia. Asphalt extender is a substitute for oil asphalt with a certain portion or not completely which still produces asphalt with characteristics that comply with standard specifications.

The utilization of this GP waste, apart from being a solution for processing waste into valuable materials, is also expected to be a solution in meeting the needs of asphalt for road construction in Indonesia, which until now, 70% of them still rely on imports (Sumantoro, 2022).

2. MATERIAL AND METHOD

2.1 Materials

Materials used in this studi is as follows:

a. Asphalt pen 60/70
b. Glycerine Pitch (GP) from the palm oleochemical industry in Bekasi Regency which is produced from the hydrolysis route

2.2 Test Method

The method used in this research is to add glycerine pitch (GP) to pen 60/70 with variations in GP content of 0%, 15%, 20%, 25%, and 30% to the weight of asphalt pen 60/70 (@3 samples) mixed using a magnetic stirrer device at a temperature of 120 °C for 15 - 20 minutes at a mixing speed of 0.4 - 0.6 kr/sec (S. Tang, 2010) until homogeneous. After being homogeneous, the asphalt from mixing GP and pen 60/70 was tested based on the basic asphalt test that has been used since the 19th century (Read and Whiteoak, 2003) namely penetration and softening point (ASTM D36, 2014) (ASTM D4-05, 2005). The maximum content that can produce asphalt with asphalt pen characteristics of 60/70 is then tested for chemical characteristics using Fourier-transform infrared spectroscopy (FTIR) (ASTM E1252, 1998) and asphalt rheology test. The FTIR test equipment used is the FTIR-Alpha-P from Bruker Optics Company with a Resolution of 4 cm^{-1}, a scanning speed of 32 sheets per second and a selected frequency range of 4000 – 500 cm^{-1}, operated by the opus software. The method for this experimental procedure is as seen in figure 1.

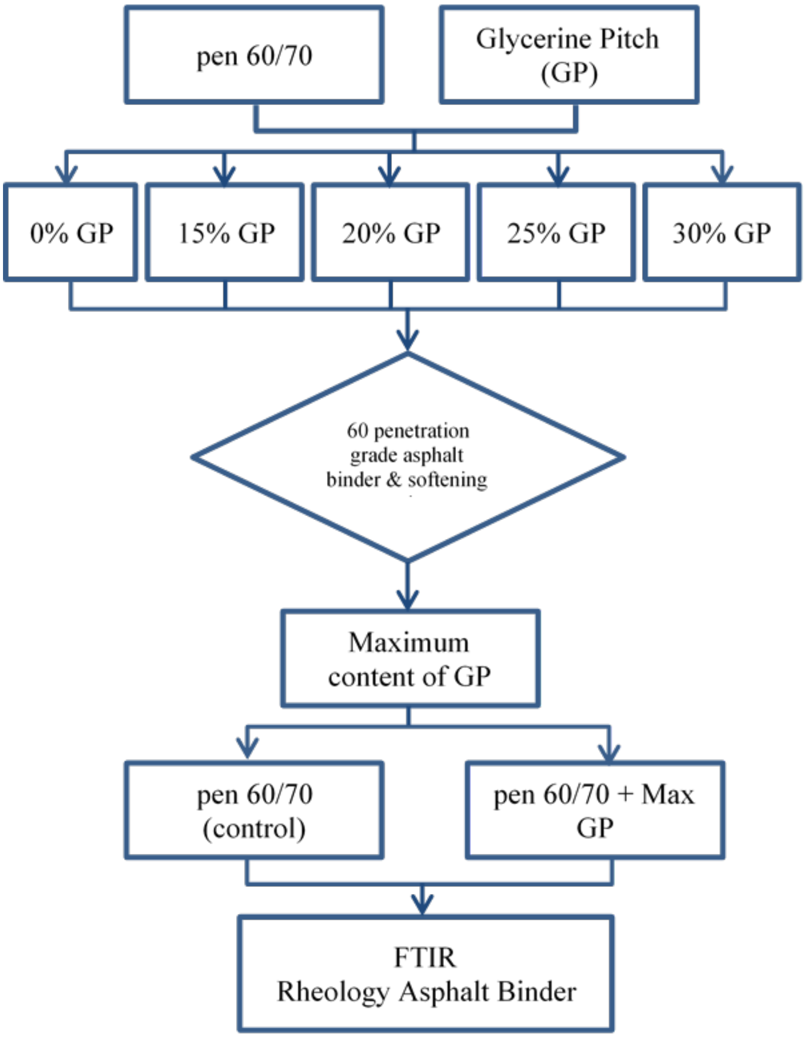

Figure 1. Flowchart of the experimental procedure

3. RESULT AND DISCUSSION

3.1 Glycerine Pitch

Before testing the effect of GP on asphalt pen 60/70, it is necessary to test the characteristics of glycerine pitch as shown in Table 1. An overview of glycerine pitch is seen in Figure 2.

Table 1. Glycerine Pitch Characteristic

Test Parameter	Unit	Result
PH	-	> 10 (Base)
Glycerol	%	13.02
Moisture content	%	2.56
Ash	%	35
MONG	%	49.42

Appearance	-	Solid brown

Figure 2. Glycerine Pitch

3.2 Asphalt Binder Extender Penetration and Softening Point

The results of adding GP to asphalt pen 60/70 are seen based on the results of penetration tests and softening points, from the results of penetration testing (Figure 3) it is known that increasing levels of GP added to asphalt pen 60/70 reduces the penetration value of asphalt, the maximum level of GP that produces asphalt with a penetration value that is still within the standard range of 60/70 asphalt pen is 25%.

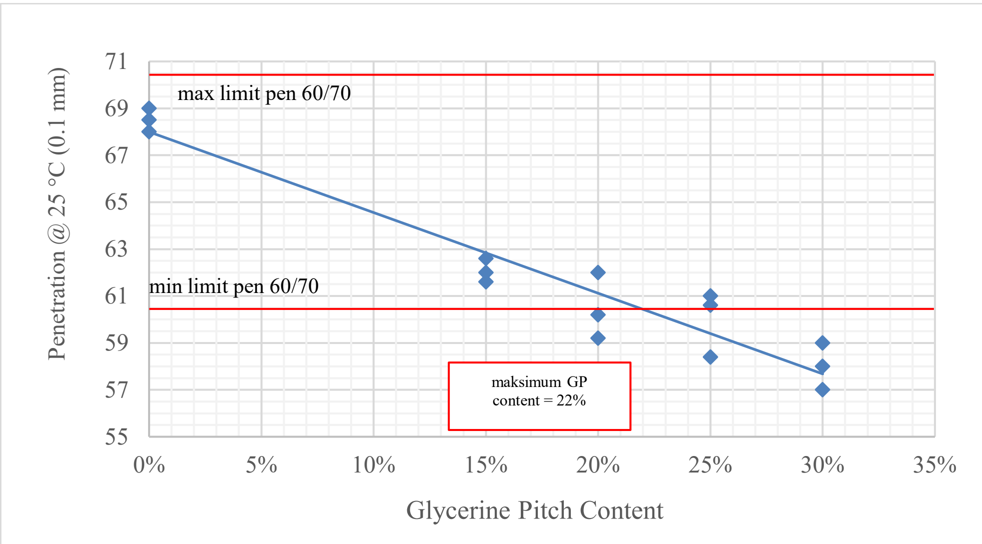

Figure 3. Effect of adding Glycerine Pitch to Asphalt pen 60/70 Penetration

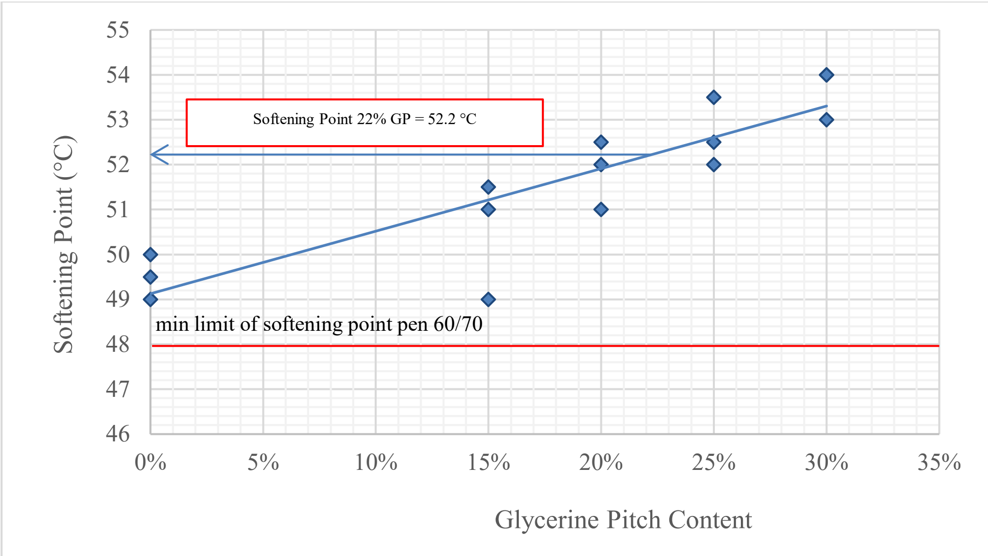

Figure 4. Effect of adding glycerine pitch to asphalt pen 60/70 softening point

While the effect on the softening point of asphalt pen 60/70, it is known that increasing levels of GP added to pen 60/70 will increase the softening point value, at 22% GP content, the resulting softening point value is 52.2 °C which is still by the standard specification for softening point for asphalt pen 60/70 is 48 °C.

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

3.3 Ashpalt Binder Extender Chemical Structure by FTIR

Chemical structure testing of asphalt was carried out using FTIR on glycerine pitch, 60/70 pen asphalt and 60/70 pen asphalt + 22% glycerine pitch. Furthermore, the test results are analyzed to identify the chemical structure of each type of asphalt which is compared with the chemical structure of pure asphaltene based on the results of the FTIR test by Leon-Bermudes and Salazar, 2008. The FTIR test results of the three types of asphalt compared to pure asphaltene are shown. in Figure 5 and Table 2.

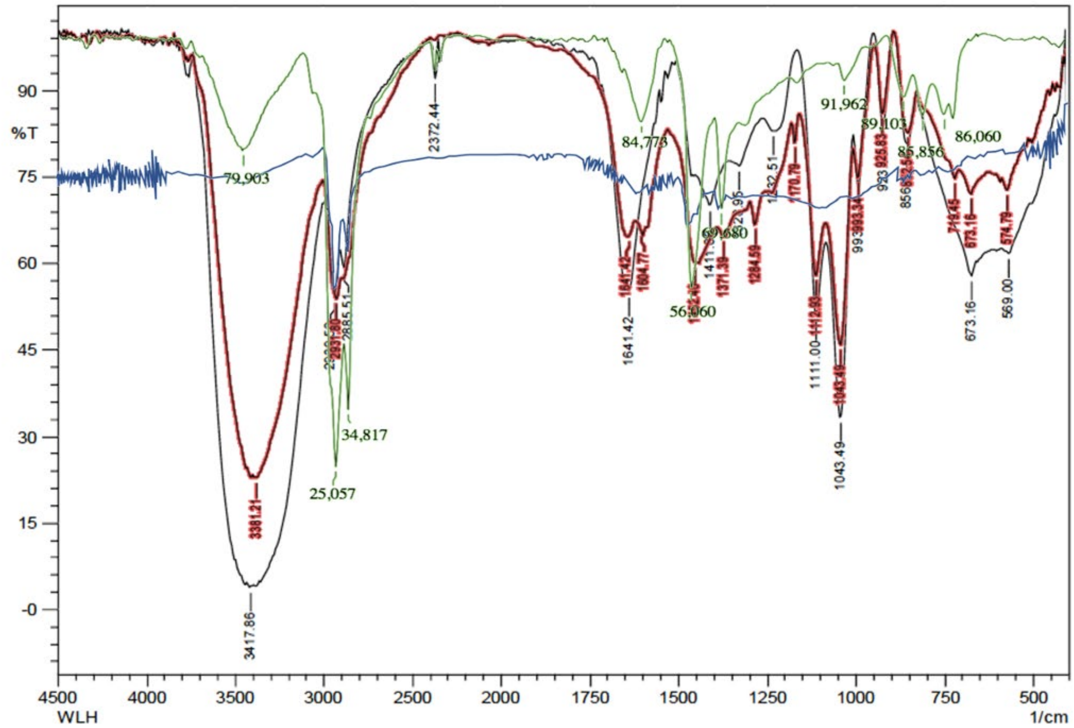

Figure 5 All samples IR spectrum

Based on the IR spectrum Table and Chart by (Skoog *et al,* 1983) and the compound functional group table by (Coates, 2006), it is known from figure 5 (green line) that asphalt binder pen 60/70 shows the -OH stretching group which identifies the presence of alcohol and phenol groups, the addition of 22% glycerine pitch increases the number of these groups. Other groups contained in asphalt pen 60/70 + 22% GP are C-H bending and C-H stretch which identifies alkanes, besides that there is also an aromatic group = $C-H^2$. Carbonyl groups are also present in the asphalt mixture and the last is the C=C group which identifies the presence of ketones, alkenes, aldehydes and carboxylic acids. The O-H stretching group increases with each addition of glycerine pitch to the asphalt, increasing the absorbance value, it is identified that compounds such as acetone, carboxylic acids, esters, and phenolic monomers evaporate during the analysis process so that more and more aromatic groups are identified. This aromatic group is very identical to most of the groups found in asphalt binder.

In table 2, it is known that the IR spectrum for pure asphaltene is entirely in pen 60/70. The result shows that pen 60/70 based on its chemical structure is asphalt. In glycerine pitch, there is 1 (one) missing IR spectrum, which is in the IR spectrum 1456 cm^{-1}. However, after GP was added to the 60/70 pen, the entire IR spectrum of pure asphaltene is found in the 60/70 + 22% GP. This indicates that the addition of GP to the 60/70 pen asphalt does not change the chemical structure of the asphalt so that GP can be used as an asphalt extender.

Table 2. IR Spectrum all samples with asphaltene

Asphaltene IR spectrum (cm^{-1})	Samples		
	Pen 60/70	Glycerine 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

3.4 Rheological Properties of Asphalt Binder Extender

Other than the chemical structure testing, the addition of GP as an extender for asphalt pen 60/70 at a level of 22% was also tested based on its rheological properties. The results of the asphalt rheological testing are shown in Table 3.

Table 3. Rheological properties of pen 60/70 and pen 60/70 + 22% GP

Test	**Unit**	**Pen 60/70 Specification**	**Pen 60/70**	**Pen 60/70 + 22% GP**
Penetration, 25 °C, 100 gr, 5 sec	0,1 mm	60 - 70	68	60
Kinematic Viscosity 135 ^{0}C	cSt	$\geq$ 300	512.62	459.96
Softening Point	°C	$\geq$ 48	50	52.2
Ductility, 25 °C, 5 cm/minute	cm	$\geq$ 100	> 100	> 100
Flash point with Clevelen Open Cup	°C	$\geq$ 232	340	325
Solubility in Trichloroethylene	%	$\geq$ 99	100.5	125
Specific gravity	-	$\geq$ 1	1.04	1.061

Based on the asphalt rheological test results shown in table 3, it is known that the addition of GP up to 22% to the weight of asphalt pen 60/70 still produces asphalt that meets the specifications of asphalt pen 60/70. This shows that glycerine pitch based on asphalt rheological test results can be used as an asphalt extender.

4. CONCLUSION

This study aims to determine the potential of glycerine pitch as an asphalt binder extender. Based on the results and discussions, it is known that:

1. Based on the results of the penetration test and softening point, the maximum content of GP as an asphalt extender is 22% of the weight of the pen anrielis860/70.
2. The chemical structure of GP shows that GP has an IR spectrum similar to asphaltene, it shows that GP has potential as asphalt, as well as after being added to asphalt pen 60/70.
3. In terms of asphalt rheology, it is known that the addition of GP as an extender still provides asphalt characteristics that are in accordance with the standard specifications of the asphalt pen 60/70.
4. Glycerine pitch based on the results of the chemical structure and rheological test of asphalt can be used as an asphalt extender.

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