CHARACTERIZATION OF ASPHALT MODIFICATION USING GROUND RUBBER AND REACTIVE RUBBER NR-g-MA AS COMPATIBILIZER

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Abstract: This study aims to improve the quality of asphalt by adding ground tyre rubber (GR) and reactive rubber (NR-g-MA) as compatibilizers of asphalt-rubber mixability. The use of GR has been widely used to improve the performance of asphalt and extend the service life of asphalt. The NR-g-MA used was the product of the copolymerization of SIR-20 rubber grafts with maleic anhydrous (MA) through a melting process in an internal mixer. Mixing of GR in asphalt was carried out in an external mixer with composition of asphalt/GR 95/5 phr at the temperature of 180^{o}C for 90 minutes. The performance of asphalts were measured based on conventional test of penetration and softening point. The rheological properties of asphalt were measured using a Dynamic Shear Rheometer (DSR). Important parameters of the rheological properties measured are complex shear modulus (G*) and phase angle (δ). Asphalt resistance to the permanent deformation (rutting) were determined on asphalt that has undergone a short-term aging process in a rolling thin film oven (RTFO) and measured at high temperatures (58-82^{O}C). Fatigue crack resistance was tested on asphalt that has undergone a long-term aging process using the method of pressure *aging vessel* (PAV) and measured at low temperatures (25-31^{O}C). Asphalt-GR mixtures' chemical, thermal and morphological properties were studied using ATR-FTIR, DSC/TGA, and SEM respectively. The results showed that adding used ground rubber (GR) decreased the penetration value by 9.93 – 18.87% and increased the softening point of asphalt from 5.57 – 11.40%. Rheological analysis showed that the addition of GR and compatibilizer NR-g-MA increase the value of complex shear modulus (G*) and decrease the phase angle (δ) of the asphalt. The addition of ground rubber and reactive rubber of NR-g-MA to asphalt improve the performance of asphalt because it has rutting resistance at high temperatures as well as improves asphalt cracking resistance at low-temperature measurements. The addition of NR-g-MA reactive rubber to modified asphalt ground rubber can increase the mixability of asphalt-rubber used tires.

Keywords: Asphalt, reactive rubber, complex shear modulus, phase angle, rutting resistance, cracking resistance

1. INTRODUCTION

1.1 Background

The increase in vehicle volume and load as well as the condition of Indonesia which has a tropical climate are the causes of the large number of damaged pavements found. The use of inappropriate asphalt often gives an indication of early damage in the form of the formation of rutting, cracks, and the rising of asphalt to the surface. Asphalt or bitumen which acts as a binder for rock aggregate used in asphalt mixtures has chemical-physical-mechanical properties that depend on changes in temperature and volume of load used (Nguyen, 2014). At high temperatures, the asphalt is viscous, melts or softens, and flexes so that it experiences plastic damage. Conversely, at low temperatures or high usage loads, asphalt becomes stiff, brittle, and easily cracked because it does not flex when receiving large loads (Moreno-Navarro, 2016). To increase the durability of the asphalt mixture and overcome the influence of external factors of temperature and pressure on the adhesive, it is necessary to modify the asphalt by adding one of the added ingredients in the form of polymer (Ma, 2020 and Merkel, 2020). Although a large number of polymer products can be used as asphalt-polymer modified additives, in general, the two groups of polymers frequently used are elastomeric and plastomer type polymers.

In previous studies, it was known that the addition of cyclic natural rubber (CNR) additives to asphalt could increase the elastic properties of asphalt, facilitate hardening, reduce penetration values, and increase the softening point temperature of asphalt (Ritonga, 2019). Besides that, increasing the elasticity of asphalt will reduce the occurrence of damage to asphalt by the mixing process at high temperatures so it will increase durability and improve the performance of asphalt mixtures (Costa, 2019). It can be understood that the addition of additives in the form of polymers will increase the complex modulus and reduce the phase angle in the asphalt mixture (Behnood, 2019). Styrenes Butadienes Styrenes (SBS) is a special and effective type of thermoplastic elastomer (TPE) as a polymer modification additive. This is because SBS has elastomeric and plastomer properties at the same time (Airey, 2014). Furthermore, Garcia-Morales (2004) has also compared the effects of adding both types of plastomer and elastomer polymer materials, namely ethylene vinyl acetate (EVA), HDPE, and SBS copolymer and concluded that asphalt added with EVA has excellent performance and has similarity with pure SBS additives and it was found that the miscibility and dispersion of EVA molecules were very good in asphalt. It was explained that the addition of 7% EVA can increase the complex modulus (G*) and loss modulus (G**) at high temperatures, which can prevent permanent damage (deformation) of asphalt. Another example of the use of polymers for asphalt modification is carried out by adding polypropylene (PP) (Casey, 2008 and Kalantar, 2012), acrylonitrile butadiene styrene (ABS) (Hasan M, 2016), polyvinyl chloride (PVC) (Arabani, 2017 and Hamedi, 2019), and polyethylene terephthalate (PET) (Leng, 2018 and Silva, 2018).

In this study, the polymer material used was used tire rubber powder (*ground tire rubber*). As it is known that the use of ground rubber for asphalt modification has also been widely reported because it is proven to be able to improve the rheological properties of asphalt mixtures (Hallmark-Haack, 2019; Sienkiewicdz, 2017 and Willis, 2012). The main problem with adding ground rubber to asphalt is its low miscibility which can affect storage stability and asphalt-rubber quality. This is due to the difference in specific gravity and the low interaction between asphalt and the rubber additives used. To overcome this problem, the addition of an appropriate matching agent to the asphalt-rubber modification was carried out. In this study, additional materials were added in the form of reactive rubber to increase the interaction between ground rubber (GR) and asphalt binder. The reactive rubber developed in this research is natural rubber copolymer-maleic anhydrous (NR-g-MA).

2. EXPERIMENTAL

2.1. Material and Methods

The binder used in this study as the base asphalt for use in modification with ground rubber was asphalt from Pertamina Inc. with penetration grade 60/70. The ground rubber (GR) was supplied by local industry which produced through mechanic grinding process, yielding a gradation of 80 mesh. The reactive rubber NR-g-MA is prepared in the melting process using an internal mixer. In this study, as much as 5% of ground rubber were added to the melted asphalt at a temperature of 180ºC, the mixture of asphalt and GR was stirred constantly for 60 minutes. For the addition of reactive rubber, NR-g-MA is added along with the addition of ground rubber (GR)

2.2. Physical Properties Test

The softening point tests and penetration tests were performed to determine the physical properties of asphalt conventionally following ASTM D36 and ASTM D5 respectively. The softening point test was performed using the ball and ring test, in accordance with ASTM D36/D36M-14. A steel ball is placed on a disk of GR-modified asphalt in a metal ring. This was placed in distilled water and heated at a rate of 5 °C/min. The softening point is the temperature when the asphalt around the steel ball has fallen a distance of 25 mm.

2.3 Rheological Testing

Rheological properties of asphalts were measured by DSR in a parallel plate configuration with a gap width of 2 mm, which is suggested for crumb rubber modified binders [24]. The asphalt sample is sandwiched between two circular plates. The bottom plate is fixed statically while the top plate oscillates back and forth across the sample at

10 rad/sec (1.59 Hz) to create a shearing action which simulates the shearing action on the asphalt pavement corresponding to a traffic speed of about 55 mph (90 km/h). The shear stress and shear strain were obtained during each cycle to calculate the complex modulus (G*), phase angle (δ), rutting resistance, and fatigue/cracking resistance in terms of AASHTO T315. The rheological properties of asphalt were tested under three aging conditions, namely original asphalt (*fresh*), short-term aging asphalt i.e asphalt samples have been treated in a hot oven called RTFO according to ASTM D2872, and long-term aging with PAV, according to ASTM D6521-13.

2.4 Thermal properties testing

Thermal properties testing was carried out using the the thermogravimetric analysis (TGA) method. The TGA used was the EXTAR/X-DSC series 700. The instrument was adopted to characterize the thermal properties of asphalt binder. A sample of about 5 mg was stored in a closed aluminum crucible at an airflow rate of 50 mL min-1 and heated at a rate of 10°C/min from room temperature to 500°C. The thermal gravimetric (TG) curve were obtained to evaluate the thermal characteristics of the asphalt binder.

2.5 Morphology Testing

Morphological testing was carried out using a tool Scanning *Elektron Microscope* (SEM) EVO® MA 10 is equipped with an Energy Dispersive X-ray (EDX) detector. SEM works on the principle of scanning an electron beam on the sample surface. The information obtained is in the form of an image created based on the detection of secondary electrons (reflected electrons) emerging from the surface of the sample. Electrons are fired at the target material so that the material that reflects the electrons will be captured by the sensor. Based on the intensity of the reflection, it will be converted into a degraded black-and-white color image.

2.6 Spectrophotometric Testing

Spectrophotometric analysis was performed with an Agilent Technologies/Cary 630 ATR-FTIR spectrophotometer over a wavenumber range of 4000 cm^{-1} up to 400 cm^{-1}, 64 scans, and 4 cm resolution^{-1}. Semi-ratio analysis of the IR absorption area of characteristic functional group peaks (e.g. carbonyl peaks) relative to polymer reference peaks (used as internal standards) to eliminate experimental errors due to film thickness variations. The spectrum obtained is stored in a computer for further analysis and manipulation. A dedicated computer is used to control the spectrophotometer with the OMNIC software.

3. RESULT AND DISCUSSION

3.1 Physical Properties

Fig.1 shows changes in physical properties, penetration, and softening points of pure asphalt and asphalt modified with ground rubber (GR) and reactive rubber (NR-g-MA). The addition of used tire rubber powder (GR) into pure asphalt decreases the asphalt penetration value and increases the asphalt softening point value. This is understandable because the addition of used tire rubber powder causes the asphalt to harden due to the presence of other particles in the asphalt. Likewise, the softening point value of asphalt increases compared to pure (blank) asphalt. The decrease in penetration value and increase in the softening point of modified asphalt samples with ground rubber are the same as the results of research conducted by Daly (2019).

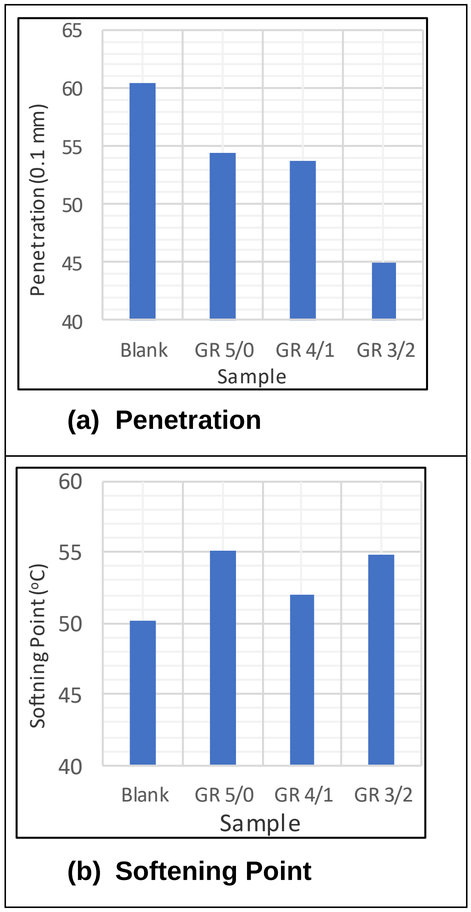


Fig.1. Penetration and softening point GR-asphalt and reactive rubber NR-g-MA.

3.2. Rheological Analysis

3.2.1 Phase Angle

As it is known that asphalt consists of constituents with low (aromatic and saturated), medium (resin), and high (asphaltene) molecular weight components. The addition of ground rubber (GR) into base asphalt (blank) changes the proportion of its component. As a result, the distribution of the molecular weight or size of the asphalt constituent components changes due to various interactions. Phase angle measurements (δ) can be used to estimate the resistance of asphalt to permanent deformation (*rutting*) and resistance to fatigue cracking on asphalt pavement. **Fig.2** presents the results of measuring the phase angle of asphalt by DSR after undergoing the RTFO aging process at the measurement temperature. As is well known, asphalt binder is a viscoelastic material, that is, it has both elastic and viscous properties, highly dependent on the measurement temperature. At low or cold temperatures, asphalt is more solid, and elastic, and has a phase angle close to 0^{O}. On the other hand, in hot or high-temperature, the asphalt material is more liquid, melts, viscous, behaves like a Newtonian fluid, and has a phase angle close to 90^{O}. It can be noted that asphalt added with 5% used tire rubber has a lower phase angle value than blank asphalt for all measurement temperatures. It is clear that the asphalt binder modified with ground rubber and compatibilizer NR-g-MA has a lower phase angle. This change occurs due to changes in the physical and chemical properties of polymer modified asphalt (Cong, 2016 and Xu, 2016)

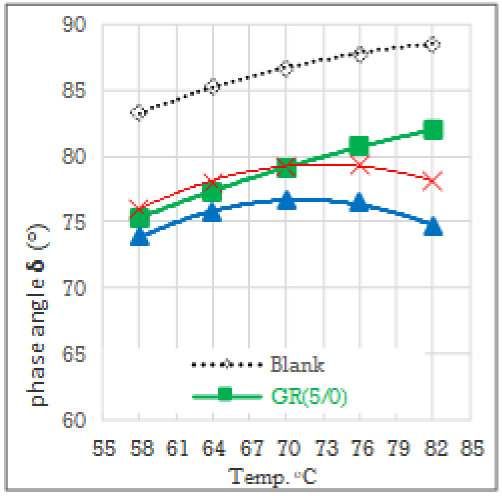


Fig.2 Phase angle of asphalt modified with GR and compatibilizer NR-g-MA.

3.2.2 Complex Shear Modulus

The complex shear modulus (G*) value of the asphalt binder describes the stiffness of the asphalt. **Fig.3** shows the complex shear modulus (G*) of the asphalts continues to decrease with an increase in measurement temperature 58 - 82^{O}C. This indicates that the material's viscous properties increase with increasing temperature measurement. It is clearly shown that the addition of ground rubber in asphalt causes the complex modulus (G*) to increase. It is understood that adding used ground tire rubber (GR) means adding an elastic component to the asphalt for each measurement temperature of 55-82°C. The addition of reactive rubber (NR-g-MA) further increases the value of G*, which means it can improve the physical and mechanical properties of asphalt. Asphalt modified with the addition of GR has much higher elastic properties than blank asphalt. The greater the value of the complex modulus (G*) the higher the elastic properties of the asphalt. Intuitively, the higher the G* value means that the asphalt is stiffer (stiffness), then the asphalt is more able to withstand loads or the greater the asphalt's ability to prevent damage (deformation).

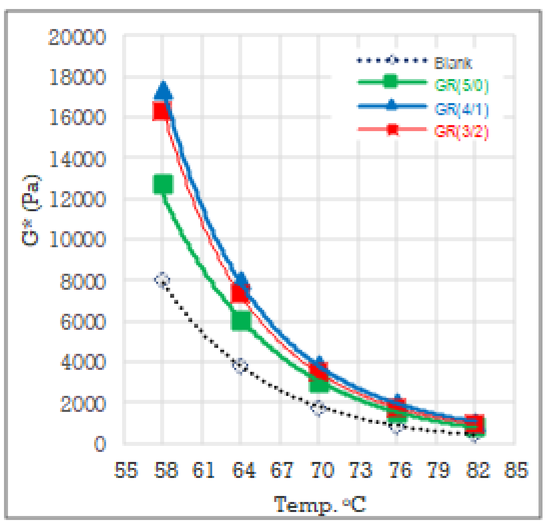


Fig.3 Complex shear modulus (G*) of asphalt with GR and reactive rubber (NR-g-MA)

3.2.3 Rutting Resistance

Permanent deformation (*rutting*) due to the low elastic properties of asphalt which is illustrated by the low value of the complex modulus (G*) of asphalt. Specification Performance Grade (PG) asphalt at high temperature is a factor that measures the stiffness of asphalt which is described in the value of G/sin δ as a parameter rutting with the basic assumption that asphalt which is more rigid and elastic can provide potential resistance to rutting. In its application, asphalt as an aggregate binder, such as in asphalt concrete, requires asphalt material that has high elastic properties or shear modulus complex (G*). The rheological data provides a quantitative measurement of the rutting parameter which is obtained by dividing the complex shear modulus G* by the sine of the phase angle (G*/sin δ) called the rutting resistance (*rutting resistance*). This factor is very important to obtain information regarding the potential for damage or permanent deformation of asphalt pavements at high temperatures. SHRP (Strategic Highway Research Program) stipulates that asphalt that has not undergone the aging process (original asphalt) must have a G∗/sin δ value equal to or greater than 1 KPa. Whereas asphalt that has undergone the RTFO aging process must have a rutting resistance value equal to or greater than 2.2 KPa. According to SHRP specifications (*Strategic Highway Research Program*) (Kennedy et al., 1994) the rutting factor allows the determination of the maximum temperature that bitumen can experience without permanent deformation. **Fig.4** shows that the G*/sin δ value of ground rubber-modified asphalt is much higher than the G*/sin δ value of blank asphalt for all temperature measurements 58-82^{O}C. The addition of 2% reactive natural rubber into asphalt has been able to increase the durability of asphalt rutting as an asphalt binding material. Asphalt added with 3% GR and 2% NR-g-MA is better because it will have a longer service life due to its higher load-bearing ability as indicated by the high G*/sin δ value after aging with RTFO. The measurements rutting resistance were also carried out by Xu et al (2016) in research on asphalt modification with rubber tires (GR) and compatible variations such as the addition of acids, oxidized polyethylene, and anhydrous polypropylene-maleic. At 70^{O}C asphalt modified with ground rubber and reactive rubber NR-g-MA has better G*/Sin δ (≥ 2.2 K.Pa) rutting resistance than blank asphalt. The results of research conducted by Xu et.al (2016) also showed that the addition of crumb rubber with various modifiers had higher rutting resistance (G*/sin δ) than blank asphalt.

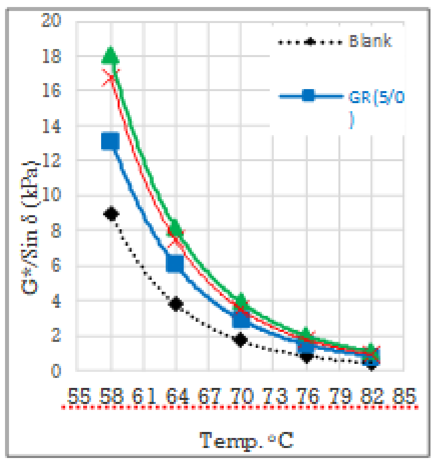


Fig.4 Rutting resistance of asphalt modified with GR and reactive rubber NR-g-MA

3.2.4 Crack Resistance

Crack resistance is one of the main characteristics of asphalt binders. For the measurement of resistance to cracking fatigue, samples that had undergone brief aging (RTFO-aged) were followed by a long aging process in high-pressure tubes at high temperatures (PAV-aged). **Fig.5** shows a comparison of the crack resistance parameters (G*.sin δ) of modified asphalt with used tire rubber powder (GR). Under the provisions of the SHRP (Strategic Highway Research Program) that asphalt that has undergone a long aging process (PAV-aged) is declared to have good quality and performance if it has a value of crack resistance parameter (G*sin δ) of less than 5000 KPa. In this study, it can be seen that pure asphalt (blank) which has been aged for a long time (PAV-aged) and rheological measurements with DSR showed a value of G*sin δ < 5000 K.Pa, meeting the maximum limit Performance Grade. It is interesting to note that with the same treatment and measurement conditions, asphalt modified with the addition of ground tire rubber (GR) has a value of G*sin.δ << 5000 K.Pa. This shows that used tire rubber powder (GR) is very effective in preventing cracks in asphalt pavements. It is stated that asphalt with a small G*sin.δ value will be able to withstand damage due to cyclic loads (cyclic loading damage).

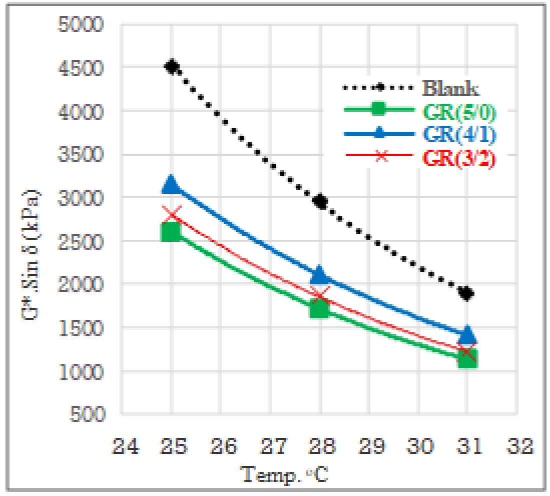


Fig.5 Crack resistance of asphalt modified with GR and reactive rubber NR-g-MA

3.3 Thermal Analysis

As a binder for road paving, the stability of asphalt against temperature changes is very important because it relates to the durability of asphalt. For this reason, it is very important to know the stability of asphalt against temperature changes. In order to examine changes in the composition of different asphalt during interaction processes with asphalt, the decomposition temperature ranges of all components in the modified sample can be obtained from the corresponding TGA graphs by analyzing the combination of the sample weight loss rate and the reported temperature. TGA curve shape profile on **Fig.6** demonstrated that asphalt loses more than 69-82 wt % when heated to 500C. The weight loss is due to the contribution of simple physical evaporation and the evolution of the remaining light components, and the occurrence of pyrolysis reactions and thermal degradation. It can be seen that the shape profile of all TG curves remains almost the same for all samples. There are three stages of decomposition that are typical for asphalt. The first stage, mass loss occurs at temperatures up to 375 ◦C, corresponding to the release of bitumen content especially saturated compounds (*saturated*) and aromatic. In the second stage, a slight variation in weight loss, and then a substantial loss occurs again in the second stage from about 375 ◦C to 425 ◦C, mainly due to the thermal degradation of the hydrocarbon materials (ie, resin and partly the bitumen fraction). The last level, degradation of residual asphaltenes and formation of charcoal residue occurred at a heating temperature of 500^{O}C with a smaller weight for modified asphalt samples. Thermal analysis study to see how the interaction between asphalt components (saturated, aromatic, resin, and asphaltene) and components of crumb rubber and other additives has been done by Yu, *et.al* (2016). Thermal analysis is very effective for measuring interactions between bitumen and various modifiers.

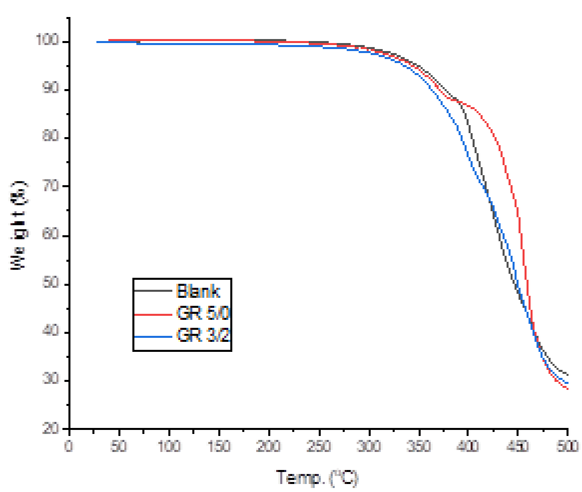
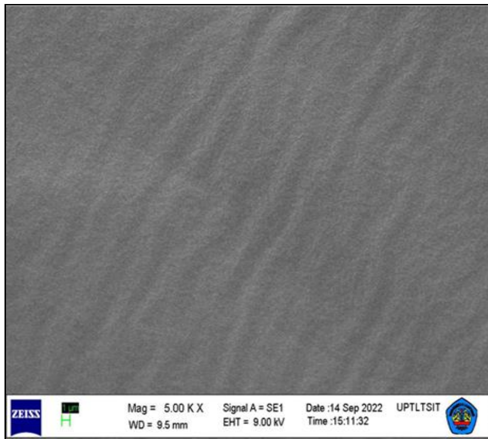


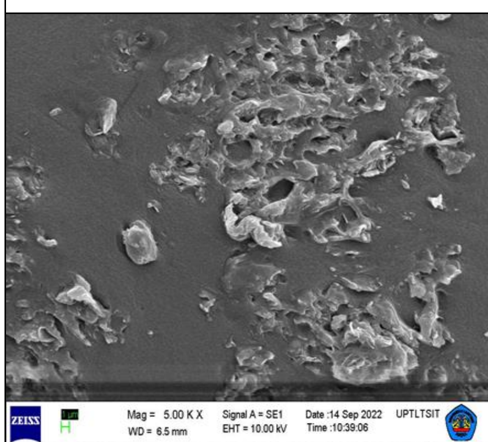
Fig.6 TGA of asphalt modified with GR and reactive rubber NR-g-MA.

3.4 Morphological Analysis

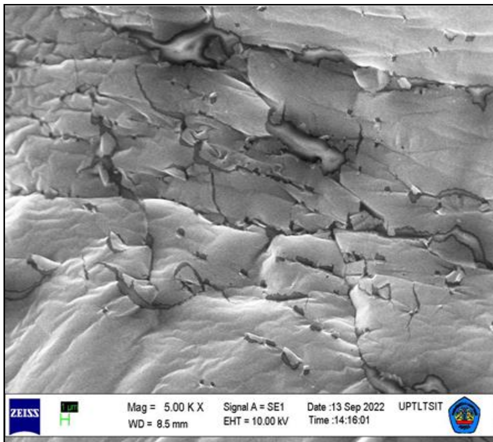
Scanning electron microscopy (SEM) of pure asphalt with a magnification of 5000 times can be seen in **Fig.7**. Pure asphalt surface looks smooth and even. When asphalt is added with 5% used tire rubber powder (GR 5/0) the asphalt surface becomes rough due to the presence of added polymer particles which are mixed unevenly. The used ground rubber is not mixed homogeneously because there is no good interaction between the asphalt molecules and the used tire rubber powder particles. Next, the addition of 2% NR-g-MA compatibilizer to the modified asphalt of used tire rubber powder (GR 3/2) makes the asphalt and used ground rubber mix better. The specific surface area of this mixture is expected to increase the stability of asphalt modified with used tire rubber powder. It can be seen that the NR-g-MA compatibilizer effectively improves the compatibility of ground rubber (GR) with asphalt.



Base Asphalt



GR 5/0



GR 3/2

Fig.7. SEM of asphalt modified with GR and reactive rubber NR-g-MA

3.5 FTIR Analysis

Fig.8 shows the infrared spectra of asphalt and ground rubber-modified asphalt. The description of the characteristics of the infrared absorption peak can be seen in Table 1.

Table 1 Description of absorption peak characteristics in Asphalt.

Absortion peak (cm^{-1})	Functional Group	Description
723	—CH2	Rocking
910 and 990	—CH=CH_2	Wagging
1027	S=O	Streching sulfoxide
1248	C—O	Stretching
1372	—CH_3	Alipatic, methyl deformation
1455, 1513	—CH_2—	Alipatik, Scissor vibration of methylene group
1591	C=C	Stretching vibration of vynil conjugation
1692	C=O	α, β-keton, stretching
1748	C=O	Stretching ester
2850	C—H	Stretching methylene asimetric
2919	C—H	Stretching methylene asimetric
3400	O—H	Stretching hidroksil
3500	N—H	Stretching amine

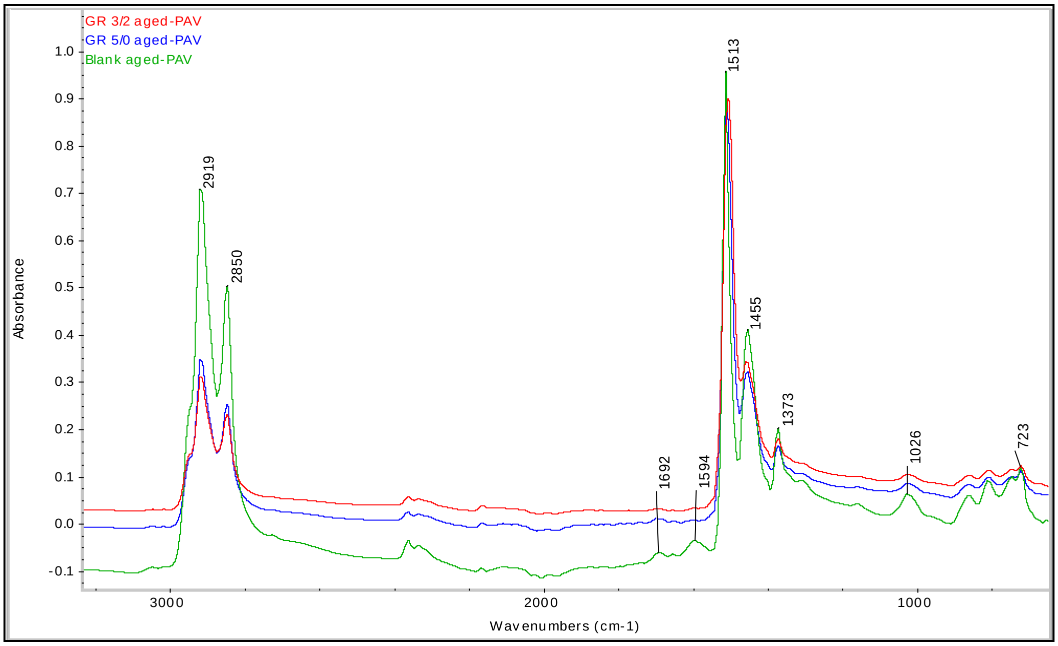


Fig.8 Spectra FTIR of asphalt modified with GR and reactive rubber NR-g-MA

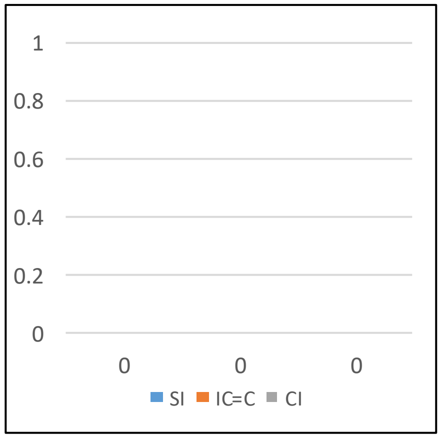
To evaluate the effect of the aging process on the asphalt binder, an absorption peak around 1600 cm^{-1} can be observed up to 1700 cm^{-1} which showed the formation of the carbonyl functional group C=O as a result of asphalt oxidation during the aging process (Feng et al., 2013; Mouillet et al., 2008). Asymmetrical deformation of C—H on $—CH_2—$ and $—CH_3$, and the symmetrical deformation C—H on vibration $—CH_3$ were observed at about 1460 cm^{-1} and 1376 cm^{-1}, respectively. The peak at 1030 cm^{-1} is ascribed to the stretching vibrations of sulfoxide (S=O). The peak that appears at 1700 cm^{-1} and 1735 cm^{-1} is the vibration of the carbonyl functional group. **Fig.8** shows that the absorption peaks of functional groups, especially oxygen-containing functional groups such as carbonyl peaks (C=O) and sulfoxide group peaks (S=O), tend to increase when the binder undergoes a long-term aging process. This is because the effects of high temperature and high air pressure change the composition of the elements and the interactions of the asphalt composite molecules. These molecular interactions include π-π interactions between aromatic rings, Van der Waals interactions between aliphatic chains, and polar interactions (hydrogen bonds, carbon bonds, ionic bonds, etc.) involving heteroatoms. As can be seen, the carbonyl peaks at 1695 and 1735 cm^{-1} increase slightly shifts to a lower wavenumber after long-term aging. In addition, the sulfoxide peaks at 1030 cm^{-1} increased significantly after aging, indicating that sulfur-containing organic compounds are sensitive to oxidation. For semi-quantitative analysis of the influence of the aging process and the degree of modification, the symmetrical deformation of C—H on vibration bonds $—CH_3$ was used as a reference absorption peak to eliminate the effect of film thickness, as the area of this peak has no correlation with either the degree of modification or the aging state. In semi-quantitative analysis, the structural indices of carbonyl groups (C=O), sulfoxide (S=O), and vinyl groups (C=C) were calculated by comparing the absorption areas of these groups with the deformation areas of the methyl groups at 1370 $cm.^{-1}$ which is used as a reference peak.

$$\text{Carbonyl Index (CI)} = \frac{A_{1865} + A_{1695\ cm^{-1}}}{A_{1370\ cm^{-1}}}$$

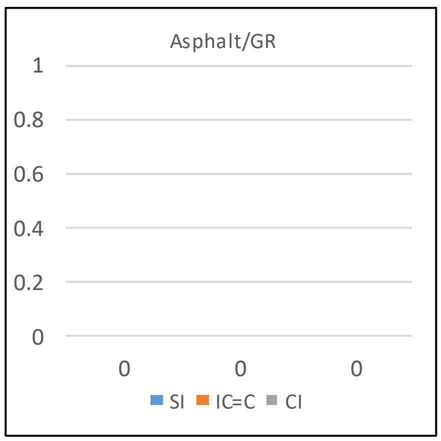
$$\text{Sulfoxide Indeks (SI)} = \frac{A_{1028\ cm^{-1}}}{A_{1370\ cm^{-1}}}$$

$$\text{Indeks C=C} = \frac{A_{1592\ cm^{-1}}}{A_{1370\ cm^{-1}}}$$

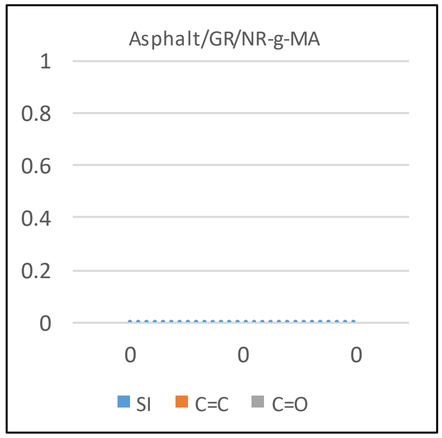
Fig.9 shows that the absorption peak intensity of the C=O carbonyl group on GR-asphalt was smaller. This shows that the level of formation of C=O carbonyl groups on asphalt modified with GR is greatly reduced. Thus, the addition of GR to asphalt can delay the oxidative aging of asphalt.



Base Asphalt (Blank)



GR 5/0



GR 3/2

Fig.9 The changes of Carbonyl, Sulfoxide, and vinyl Indexes of asphalt modified with GR and reactive rubber NR-g-MA.

CONCLUSION

Based on the above discussion, the following conclusions are obtained:

1.The addition of ground rubber (GR) in asphalt binder can improve asphalt performance as indicated by an increase in asphalt softening point and a decrease in asphalt penetration. The addition of the two polymers also increases the complex shear modulus (G*), and decreases the phase angle (δ) of the modified asphalt rheology measurement. The performance of the asphalt is increased as shown by the increased rutting resistance. This modified asphalt with ground rubber is able to prevent rutting from occurring when loaded at high temperatures.

2.To increase the mixability between asphalt and ground rubber (GR), a bridging agent is added as a compatibilizer, namely reactive rubber (NR-g-MA). The addition of this compatibilizer can increase the mixability of asphalt and ground rubber so that it can improve asphalt performance which is characterized by increasing softening point and decreasing penetration value, improving heat resistance (increasing softening point, melting point, and decomposition point), improving rutting resistance when receiving loads at high temperatures. The addition of the NR-g-MA to the asphalt/GR mixture also able to increase crack resistance. At low temperatures, asphalt/GR in the presence of NR-g-MA compatible material is more resistant to receiving loads, preventing cracking of the asphalt.

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