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Improvements in Physical and Mechanical Properties of Asphalt by Addition of Low-cost Few-layers Graphene (FLG)

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ABSTRACT: Physical and mechanical properties of asphalt have been improved by adding of few-layers graphene (FLG). FLG was obtained from a simple, low-cost and environmentally friendly liquid shear exfoliation method using a kitchen blender. The melted asphalt at temperature of 150°C was mixed with FLG at various concentrations (10 mg/ml, 20 mg/ml and 30 mg/ml) and contents (0 wt%, 3 wt%, 6 wt%, and 9 wt%) by weight of asphalt. The homogenized mixture was taken for penetration and softening point tests, while the mixing with aggregates was carried out for Marshall stability and asphalt concrete flow tests. The characteristics of void in mixture (VIM), void filled with asphalt (VFA), and void in mineral aggregate (VMA) were also investigated. The penetration values decreased (or the asphalt hardness increased) linearly with increasing of FLG concentration and FLG content. The softening point of asphalt increased as the increasing of FLG concentration and FLG content in asphalt with the average softening point increase of about 5%. The Marshall stability and asphalt concrete flow increased with increasing of FLG concentrations and FLG content. However, the addition of FLG did not affect the VIM, VFA or VMA values. Overall, the addition of FLG improves the physical and mechanical properties of asphalt and has promising prospects due to low-cost and eco-friendly nature of FLG.

Key words: Asphalt, few-layers graphene (FLG), low-cost, physical and mechanical properties

1. INTRODUCTION

Asphalts are widely used as a binding material in road construction due to their flexibility and durability [1]. Asphalts, which are one of the oldest engineering materials, are typically made from a mixture of aggregate and asphalt cement. Asphalt cement typically consists of hydrocarbons with small amounts of sulfur, oxygen, nitrogen and other trace metals [2]. The content of these chemical elements strongly influence asphalt characteristics [3,4]. One of the major challenges in the use of asphalt is its susceptibility to rutting and cracking under heavy traffic loads and extreme temperature conditions [5]. To improve the quality of asphalt, such as increasing stability, durability, water resistance, etc., it is necessary to innovate by adding additional ingredients, which are capable of overcoming the weaknesses of asphalt, for example by addition of polymer such as plastics, rubber, etc. [6,7].

Polymers are types of materials that are often used to modify asphalt, such as styrene-butadiene-styrene (SBS) or ethylene-vinyl acetate (EVA), etc. [8,9]. Polymer additives improve mechanical properties of asphalt such as elastic modulus and compressive strength [10]. Furthermore, interaction between polymer and asphalt improve the chemical properties of asphalt mixture such as thermal stability and oxidation resistance [8]. Several studies [8,11,12] have shown that mixing the polymer phase into asphalt increased the viscoelastic properties of asphalt where the asphalt mixture was resistant to thermal cracking at higher temperatures. In addition, polymer-modified asphalt concrete has better binding properties between aggregates, which leads to increased toughness [13]. Despite this, the production cost of polymer-modified asphalt is reported to be twice that of conventional asphalt [14]. Polymer additives can also cause significant changes in asphalt consistency in a relatively short period [15].

In recent years, there has been a growing interest in incorporating graphene, a carbon based nanomaterial, into asphalt to enhance its properties and performance [16–21]. It is because graphene (monolayer) have excellent mechanical and thermal properties, as well as high flexibility [17]. However, the use of graphene to modify asphalt is still rarely done by researchers considering the price is very expensive. Li et al [18] studied the properties of asphalt with graphene nanosheets (GNs), a derivative of graphene, as modifier. They showed that the incorporation of GNs (~10 layers) into asphalt effectively improved anti-aging properties, high temperature stability and temperature sensitivity of asphalt. GNs acting as asphaltene mixed physically without any chemical reactions, and the GNs behaved as a micelle nucleus evenly dispersed in asphalt. The increased viscosity due to the addition of GNs inhibited and prevented the diffusion of oxygen into the asphalt [18]. However, the commercial GNs used is expensive.

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Zhu et al. [20] studied the effect of graphene oxide (GO) addition on the hot asphalt properties. They reported that GO addition increased the viscosity of asphalt which improved the shear resistance of asphalt. Asphalt with GO addition also had a higher softening point which indicates that the asphalt was more resistant to heat damage [20]. Besides GO had good antioxidant properties, therefore it prevented asphalt from damage due to the oxidation processes. However, adding too much GO can cause high viscosity of asphalt, which can cause difficulties in the asphalt manufacturing process and application [20]. Moreno et al [21] studied about the effects of reduced graphene oxide (rGO) addition on asphalt properties. This work showed that a small amount of rGO addition (0.5%) improved thermal properties of asphalt, where the heat transfer rate increased by 34.2%. Graphene can help spread and distribute heat from the asphalt surface to the surrounding environment, thus preventing excessive heat buildup which can damage the interaction properties in the asphalt mixture [21]. However, the application of GO and rGO in asphalt mixtures is still not feasible because their prices are very high. Besides GO and rGO still contain defects that make their properties weaker than graphene.

Varrla et al. [22] made a breakthrough in the synthesis of graphene using turbulence-assisted shear exfoliation (TASE) technique in a kitchen blender. In this way, a low-cost 3~6 layers of defect-free graphene, called few layers graphene (FLG), was obtained. This study aims to investigate the effects of FLG additions on the physical and mechanical properties of asphalt. Penetration, oftening point, Marshall stability and asphalt concrete flow properties were investigated. The characteristics of void in mixture (VIM), void filled with asphalt (VFA), and void in mineral aggregate (VMA) were also examined.

2. MATERIALS AND METHOD

2.1 Materials

The materials used for asphalt production were asphalt cement with penetration 60/70 (AC pen 60/70), aggregates, and FLG. FLG was produced from graphite flakes (200 mesh), surfactant and aquadest processed in a Kenwood kitchen blender (model BL-370) [22].

2.2 Samples preperation

Asphalt that had been weighed according to the portion was heated using a stove. When the asphalt temperature reached 150ºC, then FLG was added gradually little by little to the melted asphalt while stirring for 10 minutes until the asphalt-FLG mixture was homogeneous. The amount of FLG used was varied, namely 0 wt%, 3 wt%, 6 wt%, and 9 wt% by weight of asphalt, and the concentration of FLG in asphalt was also varied, namely 10 mg/ml, 20 mg/ml and 30 mg/ml. A part of the homogenized asphalt-FLG mixture was taken for penetration test using an automatic penetrometer (ASTM D5/D5M-13) and softening point test (ASTM D36/ D36M-12). Another part of the asphalt mixture was then mixed with aggregate. This latest mixture was put into the mold and compacted with a tamper 112 times with a fall height of ± 45 cm. The compacted asphalt mixture was left for 24 hours at room temperature. Then the asphalt mixture was subjected to the Marshall test (ASTM D1559) including asphalt concrete flow value obtained using *marshall compression machine.* The characteristics of void in mixture (VIM), void filled with asphalt (VFA), and void in mineral aggregate (VMA) were also investigated. The VIM standard used was based on the provisions of road regulator in Indonesia (Bina Marga) Division 6 namely the VIM value was between 3%-5% (SNI 2439:2011), the VFA value was based on SNI 2439:2011 specification, and VMA value was based on the SNI 2439:2011 specification.

3. RESULTS AND DISCUSSION

3.1 Asphalt penetration analysis

Asphalt penetration is one of the parameters used for classifying asphalt's grade and quality for road pavement. It is a value that describes asphalt hardness at 25ºC, taken from the measurement of the depth of a standard needle that enters the asphalt after being loaded for 5 seconds. The penetration value is used to determine the asphalt's resistance from permanent deformation. The level of asphalt hardness is inversely proportional to the penetration value. Fig. 1 shows the penetration values of conventional asphalt with FLG addition in various concentrations and weight percentages in asphalt. It can be seen that, generally the penetration values decrease (or asphalt hardness increase) linearly with the increase of FLG concentration and weight percentage of FLG in asphalt. The details of asphalt hardness changes for each variation of FLG concentration and FLG content in asphalt are presented in Table 1, where the highest increase in hardness (23.7%) is shown by asphalt with the addition of 30 ml/mg FLG and 9% FLG content in asphalt. Similar trend has also been reported by Han and co-researchers for their graphene nanoplatelet (GNP) asphalt composite where the penetration decrease of about 8.4% when 0.05% GNP was added [23].

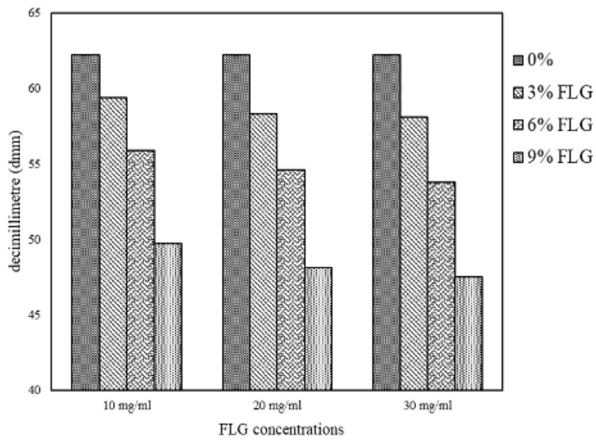

Fig. 1. Effects of FLG addition on the penetration value of conventional asphalt

The increase in asphalt hardness due to the addition of FLG changes the specifications of asphalt pen 60/70 to asphalt pen 40/50, a hard asphalt that can be used as asphalt for highways with high traffic volumes and in tropical areas. According to Wang, et al [24] when the asphalt-FLG composite is under pressure, the graphene layers will distribute the pressure in all directions, therefore the penetration of the penetrometer needle in the asphalt decreases. However, this is not recommended because if too much graphene is added, the penetration value obtained will be too small, thus it does not match the standard value (SNI 2456: 2011).

Table 1. The penetration's decreases (or asphalt hardness increases) in percentage of each asphalt after FLG addition.

FLG Concentration (mg/ml)	Weight Ratio of FLG in asphalt		
	3%	6%	9%
10	4.5	10.1	20.1
20	6.3	12.2	22.7
30	6.6	13.5	23.7

3.2 Effect of FLG addition on the softening point of asphalt

Asphalt's softening point or asphalt melting point is one of the main parameters for classifying asphalt grade and quality for road pavement. This empirical test is the main approach besides asphalt penetration used to determine asphalt resistance to permanent deformation. The softening point examination aims to measure the temperature value at which the steel balls push down the asphalt layer on the ring, until it touches the base of the plate located under the ring at a distance of 1 inch, as a result of a certain heating acceleration [25]. This examination is needed to determine the resistance limit of bitumen asphalt to the temperature changes after the addition of FLG.

Fig. 2 shows the softening point temperatures of asphalt with and without the addition of FLG in various concentrations and percentages in asphalt. Generally, it can be seen that the softening point temperatures of asphalt increase with the increasing FLG concentrations and FLG contents in asphalt. The increase of asphalt-FLG softening point towards the softening point of asphalt without the addition of FLG averagely is about 5%. Seeing this trend, it is estimated that the softening point of the asphalt-FLG composite will continue to increase if the concentration and amount of FLG in the asphalt are continuously added, but with a smaller increase. A similar trend is also reported by Moreno et al [21] for their asphalt - reduced graphene oxide (rGO) composites. As the graphene content increases in asphalt, which leads to an increase in the softening point of asphalt, the susceptibility of asphalt to high temperatures will decrease [21]. This is due to the character of graphene, in this case FLG, as a good heat conductor which causes asphalt to transfer heat quickly and evenly to all parts of the asphalt body.

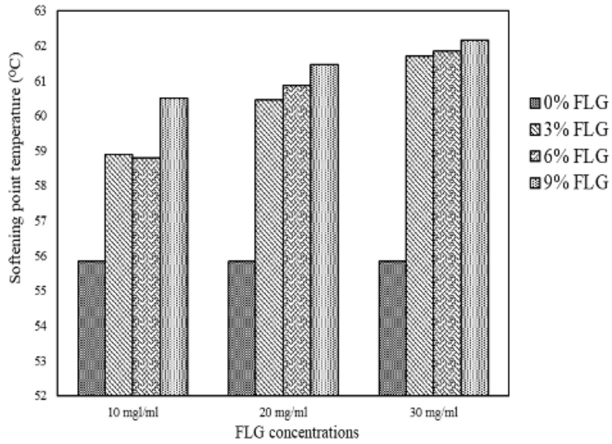

Fig. 2. Effects of FLG addition on the asphalt softening point

Zhou et al [26] conducted molecular modeling and simulation regarding the thermo-mechanical properties of the interaction between graphene/carbon nanotubes and asphalt bituminous asphalt. They reported the presence of intermolecular attractions (secondary bonds) between bituminous asphalt and graphene. This secondary bond resulted an increase in the softening point of asphalt due to stronger bonds between materials that affected their physical properties such as melting or boiling point [26].

3.3 Effect of FLG addition on the Marshall stability and asphalt concrete flow

Marshall stability is a measure of the static strength of asphalt mixtures used in road construction. This test measures the ability of an asphalt mixture to withstand loads without significant cracking or damage [27]. This test is carried out using a machine that packs an asphalt sample with a specified load and measures the resulting compressive strength. A higher Marshall stability value indicates a stronger and more stable asphalt mixture [28]. Fig. 3 shows the Marshall stability values versus FLG addition in various concentrations and contents. Generally, Marshall stability values increase with increasing FLG contents and concentrations. The increases of Marshall stability compared to the conventional asphalt stability without FLG addition (1151.2 kg) are about 5.1% to 17.6% when 3 to 9% FLG are added at concentrations of 10 to 30 mg/ml. Eisa, et. al [29] reported that their graphene nanoplatelets (GNP) could increase the Marshall stability value of asphalt because it has strong mechanical properties and is resistant to pressure. In line with this, FLG can provide additional strength and stability to the asphalt matrix, thereby increasing the ability of asphalt to withstand loads and reducing susceptibility to damage.

There are at least three mechanisms of FLG interaction in asphalt. First, FLG adheres to the asphalt aggregate surface and forms a thin film that increases the strength and stability of the asphalt matrix. Second, FLG is dispersed in the asphalt matrix and provides additional strength and stability at the molecular scale. Third, graphene can interact with chemical compounds in asphalt such as bituminous acid in terms of secondary bonds improving the mechanical and electrical properties of asphalt.

Regarding the asphalt concrete flow, it is the amount of change in shape (plastically) of an asphalt mixture that occurs as a result of being subjected to a load until failure occurs, expressed in units of length [30]. Flow in Marshall test terminology is the amount of vertical deformation that occurs from the start of loading until the stability condition begins to decrease [31]. Fig. 4 shows the relationship between the content and concentration of FLG in asphalt on the asphalt concrete flow properties. The figure shows that the asphalt concrete flow value increases with the increase in FLG content, with an average increase of around 3.5% to 17.7% compared to conventional asphalt without the addition of FLG which has a flow value of 3.8 mm. This means that the addition of FLG causes an increase in the elasticity of asphalt. It can be explained that the addition of graphene to asphalt can improve the mechanical properties of asphalt, such as resistance to pressure and friction. This is because graphene can strengthen the bonds between asphalt molecules. In addition, graphene can also increase the thermal stability of asphalt, thereby slowing down the aging process and the development of cracks on the road surface [21].

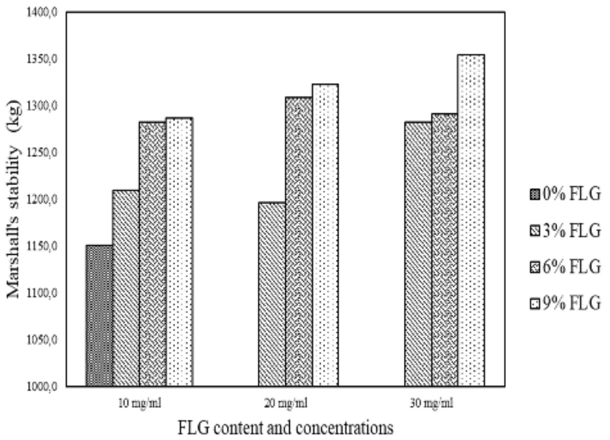

Fig. 3. Effects of FLG addition on the Marshall stability of asphalt.

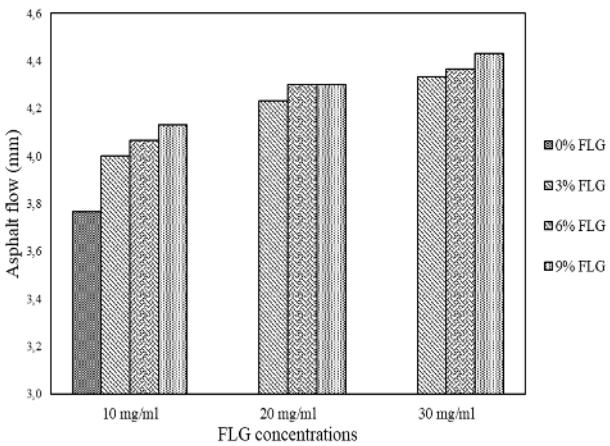

Fig. 4. Effects of FLG addition on the asphalt concrete flow.

3.4 Effect of FLG addition on void in mixture, void filled with asphalt, and void in mineral aggregates

Void in mixture (VIM) is the volume of pores remaining after a mixture of asphalt concrete is compacted. Too large VIM will reduce the water tightness of asphalt increasing the asphalt oxidation process which can accelerate asphalt aging and reduce the durability properties of asphalt. However, a too small value of VIM will cause pavement bleeding when the temperature increases. Factors affecting the VIM value are the asphalt content added, the coarse form and the medium used. VIM is expressed as a percentage of the volume of asphalt concrete. Fig. 5 shows the effect of FLG addition on the VIM value. It can be seen that the addition of FLG can reduce the VIM value even though it is not significant, so it can be said that the addition of FLG generally does not affect the VIM value.

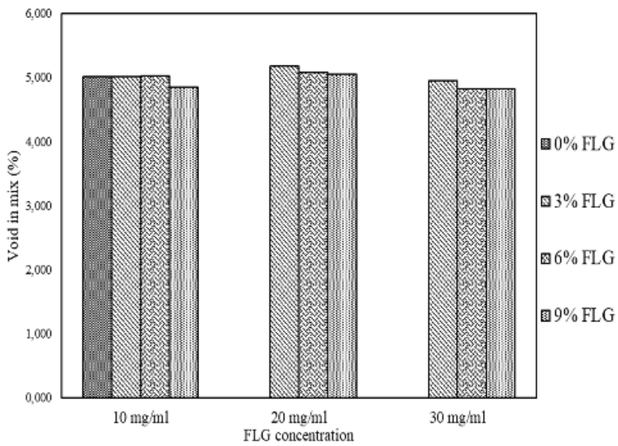

Fig. 5. Effect of FLG addition on void in mix of asphalt

Void filled with asphalt (VFA) is the percentage of voids among aggregate particles that have been filled with asphalt [32]. Filling voids is useful to increase the stability and strength of the road, as well as to reduce erosion and water damage. Besides that, void filling is also useful for improving road performance during extreme weather, such as rain and snow. A high VFA value indicates the number of cavities that have been filled with asphalt so that the mixture becomes more water-air tight. Conversely, if the VFA value is small, it will cause the mixture to be less impermeable to water and air, so that the asphalt mixture is easily oxidized which eventually causes the pavement layer to deteriorate quickly. The value of VFA is affected by asphalt content, aggregate gradation, number of collisions and compaction temperature. According to SNI 2439:2011, the minimum VFA value is 65%. Fig. 6 shows the effect of FLG addition on the VFA value. It can be seen that the VFA value of asphalt with FLG addition experienced a slight increase or decrease (fluctuation) compared to asphalt without FLG addition (67.1%). Even so, it can be said that there is relatively no change in the VFA value when the asphalt is added with graphene. This is possible considering that FLG is physically very small on the nanometer scale.

Voids in mineral aggregate (VMA) is the pore volume in a solid asphalt mixture if the entire asphalt's blanket is removed. Pore volume in internal aggregates itself is excelueded. VMA is expressed as a percentage in a hot asphalt mixture. VMA value was influenced by asphalt content, aggregate gradation, the number of collisions and compaction temperature. The small VMA causes the asphalt covering the aggregate to be limited, leading the pavement to suffer damage quickly. Fig. 3.7 shows the effect of FLG addition on the VMA value of asphalt. It can be seen that the VMA value of asphalt-FLG composite increased and decreased (fluctuated) but not significantly compared to asphalt without the addition of FLG (about 15.6%). Thus, the contribution of FLG to the value of VMA is insignificant. Nevertheless, all test results show a VMA value that meets the standard specifications of SNI 2439:2011 where the minimum VMA value is 14%.

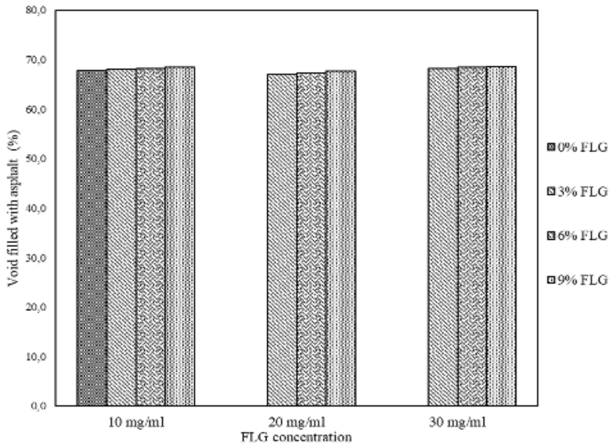

Fig. 6 Effect of FLG addition on void filed with asphalt (VFA)

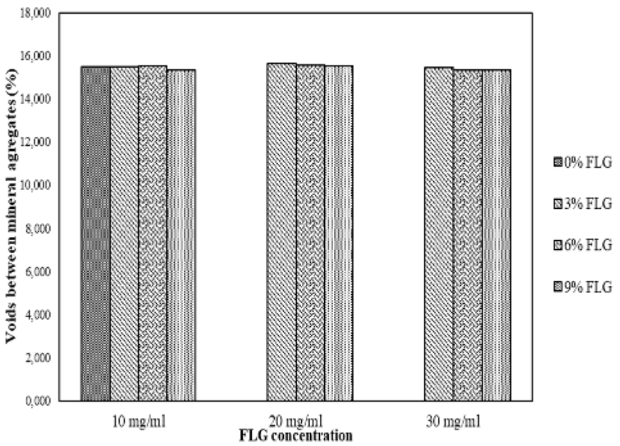

Fig. 7 Effect of FLG addition on VMA value

4. CONCLUSIONS

The physical and mechanical properties of asphalt with the addition of few-layer graphene (FLG) have been investigated. The penetration values decreased (or the asphalt hardness increased) linearly with increasing FLG concentration and FLG content in asphalt. The highest increase in hardness (23.7%) was shown by asphalt with the addition of FLG at a concentration of 30 ml/mg and an FLG content of 9% by weight of asphalt. The softening point of asphalt increased with increasing FLG concentration and content. The average softening point increase was about 5% after the FLG addition. Marshall stability values increased with FLG contents and concentrations by about 5.1% to 17.6% when 3 to 9% FLG were added at concentrations of 10 to 30 mg/ml. Asphalt concrete flow increased with the FLG content, with an average increase of around 3.5% to 17.7%. Overall, the addition of FLG improved the physical and mechanical properties of asphalt and had promising prospects due to low-cost and eco-friendly nature of FLG.

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