

MODIFICATION OF ASPHALT BINDER USING BIO-OIL AND BIO-CHAR  
FROM OIL PALM BIOMASS

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MODIFICATION OF ASPHALT BINDER USING BIO-OIL AND BIO-CHAR  
FROM OIL PALM BIOMASS

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## DEDICATION

This thesis is dedicated to my beloved children,

*Dina, Dania, Tuah & Haikal*

You have made me stronger, better and more fulfilled than I could have ever  
imagined.

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## ABSTRACT

Agriculture biomass is found to be a potential alternative to petroleum asphalt binder used in pavement due to its carbon nature and morphology. Efforts have been made to look for practical applications of biomass. Bio-oil (BO) and bio-char (BC) derived from biomass of oil palm empty fruit bunches (EFB) could produce a wide range of products including energy source, chemicals and raw materials. Therefore, the aim of this study is to investigate the performance of asphalt binders modified by BO and BC generated from oil palm EFB biomass. In the first phase, pyrolysis process was carried out to produce BO and BC from oil palm EFB biomass. A new laboratory-scale machine has been designed for this purpose. BO and BC were characterized based on density test and elemental analysis. In the second phase, these additives were incorporated into 80-100 PEN asphalt binder at different percentages (0%, 5%, 10% and 15%) in order to obtain the recommended additive content. All samples were tested in the laboratory for their physical, rheological and chemical properties. Penetration, softening point, viscosity, storage stability, dynamic shear rheometer (DSR) and Fourier transform infrared (FTIR) tests were performed on the modified asphalt binder. The recommended amount of 5% BO and 5% BC were then be used in phase three for two types of asphaltic concrete mixtures. Mixtures with 14 mm nominal maximum aggregate size (AC 14) consisting 100% fresh aggregate (fresh mixture) and 60% fresh aggregate incorporated 40% reclaimed asphalt pavement (RAP) aggregate (RAP mixture) were tested. The performance tests include resilient modulus, dynamic creep and indirect tensile strength tests. Based on the findings, addition of BC shows 14% increment in rutting and fatigue resistance performance of fresh mixture. However, no significant improvement on performance of RAP mixture with the addition of BO and BC.

## ABSTRAK

Biomass pertanian didapati berpotensi sebagai alternatif kepada pengikat asfalt petroleum yang digunakan dalam turapan kerana sifat dan morfologi karbonnya. Usaha-usaha telah dijalankan untuk mencari aplikasi biomass yang praktikal. Bio-oil (BO) dan bio-char (BC) yang berasal daripada biomass tandan buah kosong (EFB) dapat menghasilkan pelbagai produk termasuk sumber tenaga, bahan kimia dan bahan mentah. Oleh itu, kajian ini bertujuan untuk menyiasat prestasi pengikat asfalt yang diubahsuai dengan BO dan BC dihasilkan daripada biomass EFB kelapa sawit. Dalam fasa pertama, proses pirolisis telah dijalankan untuk menghasilkan BO dan BC daripada biomass EFB kelapa sawit. Satu mesin baru berskala makmal telah direkabentuk untuk tujuan ini. BO dan BC telah dicirikan berdasarkan ujian ketumpatan dan analisis elemen. Dalam fasa kedua, bahan-bahan tambah ini telah digabungkan ke dalam pengikat asfalt 80-100 PEN pada peratusan yang berbeza (0%, 5%, 10% dan 15%) untuk mendapatkan kandungan bahan tambah yang dicadangkan. Semua sampel telah diuji di dalam makmal untuk mendapatkan ciri-ciri fizikal, reologi dan kimia. Ujian penusukan, titik lembut, kelikatan, kestabilan storan, *dynamic shear rheometer* (DSR) dan fourier transform infrared (FTIR) telah dijalankan ke atas pengikat asfalt terubahsuai. Kandungan 5% BO dan 5% BC yang dicadangkan kemudian digunakan pada fasa tiga untuk dua jenis campuran konkrit berasfalt. Campuran dengan saiz agregat nominal maksimum 14 mm (AC 14) yang mengandungi 100% agregat segar (campuran segar) dan 60% agregat segar digabungkan dengan 40% agregat turapan asfalt tebusguna (RAP) (campuran RAP) telah diuji. Ujian prestasi termasuklah ujian modulus kebingkasan, rayapan dinamik dan kekuatan tegangan tidak langsung. Berdasarkan dapatan kajian, penambahan BC menunjukkan peningkatan 14% dalam prestasi rintangan terhadap aluran dan kelesuan campuran segar. Namun, tidak ada peningkatan yang ketara terhadap prestasi campuran RAP dengan penambahan BO dan BC.

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## LIST OF ABBREVIATIONS

AASHTO	-	American Association of State Highway and Transportation Official
AC 14	-	Asphalt concrete of nominal maximum aggregate size 14 mm
ASTM	-	American Society for Testing and Material
ATR	-	Attenuated Total Reflection
BO	-	Bio-Oil
BC	-	Bio-Char
CHNS	-	Carbon, hydrogen, nitrogen, and sulphur
COPF	-	Cellulose oil palm fibre
DSR	-	Dynamic Shear Rheometer
EFB	-	Empty fruit bunch
EVA	-	Ethylene Vinyl Acetate
FTIR	-	Fourier-Transform Infrared
HHV	-	Higher heating value
HMA	-	Hot Mix Asphalt
ITS	-	Indirect tensile strength
JKR	-	Jabatan Kerja Raya
LTA	-	Long-term aged
OAC	-	Optimum asphalt content
OPC	-	Ordinary Portland Cement
PAV	-	Pressure aging vessel
PI	-	Penetration index
POFA	-	Palm oil fuel ash
PKO-p	-	Palm kernel oil polyol
RAP	-	Reclaimed asphalt pavement
RTFO	-	Rolling thin film oven
SARA	-	Saturates, aromatics, resins and asphaltene
SBS	-	Styrene-butadiene styrene
SBR	-	Styrene-butadiene rubber
SHRP	-	Strategic Highway Research Program

SMA	-	Stone mastic asphalt
STA	-	Short-term aged
TMD	-	Theoretical specific density
UTM	-	Universal Testing Machine
VFB	-	Voids filled with bitumen
VTM	-	Voids in total mix

## LIST OF SYMBOLS

$G_{se}$	-	Effective specific gravity of aggregate
$G_{sb}$	-	Bulk specific gravity of aggregate
$G^*$	-	Complex modulus
$\delta$	-	Phase angle
$G_{mm}$	-	Maximum specific gravity of the mixture
$I_{C=O}$	-	Carbonyl oxidation index
$I_{S=O}$	-	Sulfoxide oxidation index
$S_t$	-	Indirect tensile strength, kPa
$M_R$	-	Resilient modulus, MPa
$E$	-	Creep stiffness modulus, MPa

# CHAPTER 1

## INTRODUCTION

### 1.1 Background of Research

Asphalt pavement is a means of transportation that contributes to economic growth and development and social benefits. Most of the world's pavement surfaces are made of flexible pavement constructed from asphalt surface using HMA design (Pan, 2013). The asphalt is heated with a combination of coarse and fine aggregates using HMA procedures. However, an increase in the traffic load every year has contributed to pavement distress, requiring higher maintenance to prevent further damage. As a developed country, Malaysia has experienced an increase in individual demand for motor vehicles, which contributes the road distress with higher road maintenance. Since 1976, the Public Works Department (PWD), under the Ministry of Works Malaysia (MOW), has allocated millions of Ringgit Malaysia (RM) to carry out maintenance for federal and state roads. Figure 1.1 shows that allocation for road maintenance had increased by billion RM annually from 1995 to 2019 (JKR, 2019). By the end of 2019, there were 31.2 million vehicles, including personal cars, commercial vehicles, public vehicles and motorcycles registered in Malaysia, as compared to 20 million in 2009 (JPJ, 2016; Lim, 2020) as shown in Figure 1.2. Generally, ruts may occur on the pavement's wheel path as a result of the traffic load. The increasing axle load repetition on the pavement surface has been a major contributor to fatigue failure (Igwe, 2014).

For over a decade, many pavement researches have been conducted to reduce pavement distress through a mix design and asphalt binder. The use of modifier or additive in asphalt binders and mixture is one of the ways to improve the hot mix asphalt (HMA) performance. At the same time, researchers need to consider that the world's fuel fossil resources are decreasing. As known, the asphalt binders used in the flexible pavement are derived from distillation of petroleum classified as fossil fuel

(Gong et al., 2016). Fossil fuels such as coal, gas and petroleum are considered as non-renewal energy sources. Thus, non-renewal energy comes from sources that will not be replenished for thousands or millions of years. Fossil fuels, which contain high carbon content, have become the world's leading source of energy. The wide-scale use of fossil fuels has prompted scientists to develop renewable energy resources. Renewable energy such as oxygen, freshwater, solar energy, timber and biomass can be repeatedly used since it is replenished by the environment,

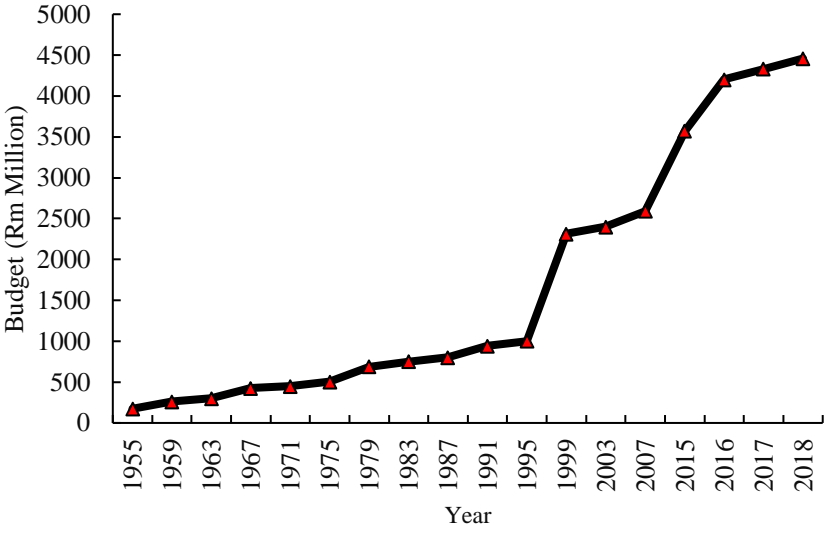


Figure 1.1 State roads maintenance allocation (JKR, 2019)

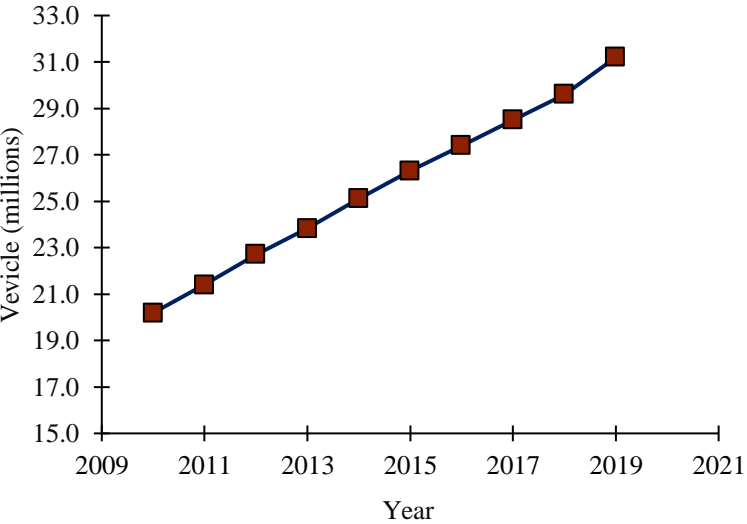


Figure 1.2 Number of registered vehicles in Malaysia (JPJ, 2016; Lim, 2020)



For this reason, asphalt researchers are very concerned about this issue where various studies have been carried out on the combination of biomass from agriculture as modifier or filler in HMA. The utilization of bio-binders derived from biomass could offer cheaper and easily available materials for the existing bitumen industry to deal with current issues such as exhaustion of crude oil reserves and fluctuation in global crude oil prices. The char from biomass has been commonly used in modification of mixture performance. In the early days, the utilization of biomass focused on the generation of common combustion-based heat. Recently, researchers have grown more interested in the use of the thermal conversion method using pyrolysis process heating at high temperature yield different types of by-product such as solid (bio-char) gaseous, and liquid fuels (bio-oil) (Cai et al., 2014).

Based on the IEA Bioenergy Countries' Report (2018), Finland is ranked first of solid biomass with the highest (up to 63 GJ/capita) energy consumption followed by Sweden, Austria, Denmark. It was also noted that biomass was specifically used for energy purpose activities. Meanwhile, Malaysia also generated a large amount of biomass from agricultural waste including paddy, woody, municipal waste, oil palm and others as shown in Figure 1.3. The oil palm plantations contributed the biggest amount of biomass from agriculture. The biomass which is produced from the oil palm industries include biomass i.e. oil palm trunks (OPT), oil palm fronds (OPF), empty fruit bunches (EFB) and palm pressed fibres (PPF), palm shells and palm oil mill effluent (POME) (Ani et al., 2014). However, the EFB biomass shows the large potential for transforming EFB into useful bio-product for energy. In 2014, 7.34 Mt (million metric ton) of processed oil palm EFB had produced an estimated total of 40.55 Mt oil palm biomass (Loh, 2017). For this reason, previous study has focused on the use of oil palm biomass on the asphalt performance. Although it is difficult to find other materials that can meet specific asphalt requirement as an alternative binder, biomass have drawn attention to carbonaceous asphalt.

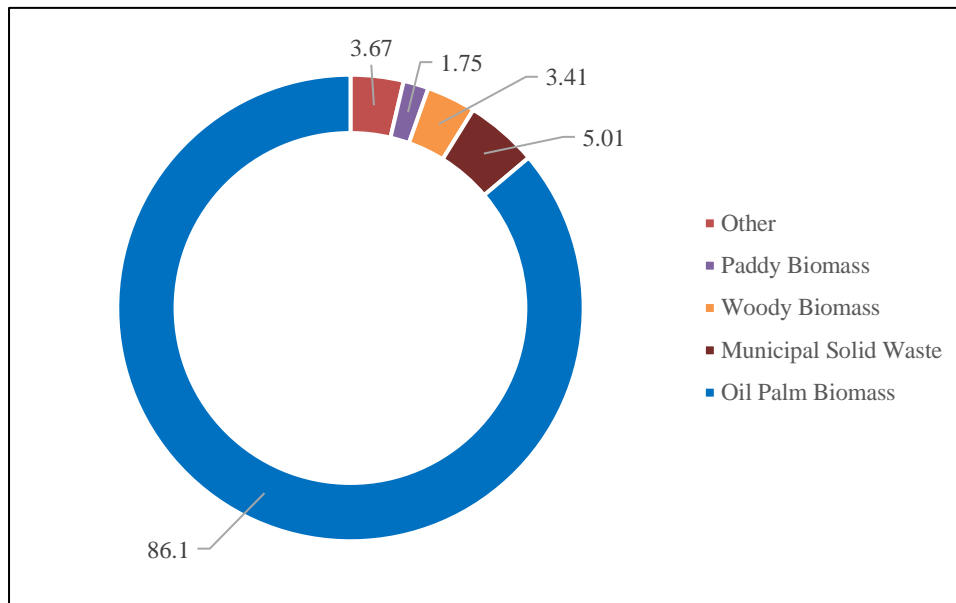


Figure 1.3 Biomass sources in Malaysia (How et al., 2019)

## 1.2 Problem Statement

Recently, many experts in petroleum and pavement industry have explored the new alternative resources to reduce dependency on petroleum products. Likewise, the pavement industry urgently needed to improve the service life of road surfaces through asphalt innovations and technologies. Using innovative materials for asphalt is expected to give advantages in quality, cost and convenience. The bio-oil (BO) and bio-char (BC) from the EFB from oil palm biomass show many potential opportunities to produce multiproduct for energy. BO and BC achieved higher carbon content after the pyrolysis process, showing that it could be more compatible with asphalt after the modification process. However, limited studies on pyrolysis products of EFB biomass (BO and BC) to improve the asphalt performance have been conducted.

Most previous studies focused on using non-pyrolyzed from oil palm biomass such as palm oil fuel ash (POFA) and cellulose oil palm fibre (COPF) as modified asphalt binders rather than a mixture. Thus, the asphalt's physical properties were improved using oil palm biomass from the base binder (Rusbintardjo et al., 2014; Sukaimi et al., 2018). However, these studies have not discovered the performance of

rheological and chemical properties. The oxidation of asphalt during the aging process may change the concentrations of chemical compositions. Additionally, the rheological properties of asphalt are depended on the chemical properties of asphalt. It is important to ensure that oil palm biomass is used as anti-oxidants in order to maintain the chemical compositions in asphalt.

Past researchers also found that using pyrolysis products from other agriculture biomass as a modified binder has improved or is similar to the conventional binder (Yang et al., 2017; Zhao et al., 2014). Therefore, these issues have motivated this research to accept the challenge of using empty fruit bunch biomass to improvise the performance of asphalt mixture. Also, issues on higher pavement distress are always getting criticisms from public perception towards the government and stakeholders. It is also expected that the findings can be used as fundamental guidelines and recommendations in future research for a good alternative biomass based binder.

### **1.3 Objective**

The aim of this research is to investigate pyrolysis products (BO and BC) obtained from EFB biomass as modifiers for asphalt binder. The specific objectives of this study are as follows:

- (a) To produce and characterize BO and BC from EFB biomass using fast pyrolysis method.
- (b) To determine the physical, rheological and chemical properties of BO and BC modified asphalt binders.
- (c) To determine the optimum BO and BC content in the modification of asphalt binder
- (d) To evaluate the effect of BO and BC modified asphalt binders on the performance of HMA mixture

## **1.4 Scope of Research**

This research focuses on laboratory investigations on the effectiveness of adding pyrolysis materials, such as BO and BC, into base asphalt binder. Fast pyrolysis process by using rotary pyrolyzer was conducted to extract the BO and BC from EFB biomass. Extracted BO and BC were used to modify fresh asphalt (80-100 PEN) with the addition of 5%, 10% and 15% (by weight of asphalt). Properties of modified asphalt binder from BO and BC were compared with the unmodified asphalt binder. The properties were evaluated based on laboratory property tests, including short and long-term aging processes. The suitable modified asphalt binder from bio-oil and bio-char were applied on the mixture gradation AC 14 in accordance with JKR (JKR, 2008). Two types of mixture gradation were used, which were from fresh aggregates and recycling aggregates from reclaimed asphalt pavement (RAP). The Marshall test procedure and volumetric analysis will be used in the preparation of the sample specimen. Performances of the mixtures were also evaluated based on the laboratory sample and test i.e., dynamic creep test, indirect tensile test and resilient modulus and test i.e. dynamic creep test, indirect tensile test and resilient modulus.

## **1.5 Significance of Research**

The findings of this study will be beneficial in the following ways:

- (a) Facilitate the promotion of pyrolysis product from EFB biomass as the modifier for asphalt
- (b) As an alternative binder developed from agricultural waste and to highlight the diversity in EFB usage
- (c) Improve the asphalt binder properties of asphalt mixtures performance in a sustainable and economical way
- (d) Assist in developing value-added products from local resources to promote carbonaceous in the pavement industry
- (e) Provide significant market value for road agencies and municipalities in Malaysia

## **1.6 Thesis Outline**

This thesis is organized into five chapters and the outlines are briefly summarized as follows:

CHAPTER 1: Briefly describes the research perspectives, including background, problem statement, aim and objectives.

CHAPTER 2: Provides a literature review of previous research, especially the use of EFB biomass waste for asphalt binder, research work experience in a conducted experiment and HMA performance.

CHAPTER 3: Presents in detail the processes involved in preparing HMA pyrolyzed materials and detailed methodology of the laboratory testing.

CHAPTER 4: Discusses the experimental findings based on the methodology described in Chapter 3.

CHAPTER 5: Provides conclusions and recommendations based on the results obtained.

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## LIST OF PUBLICATIONS

- (a) Poh, C. C., Hassan, N. A., **Raman, N. A. A.**, Shukry, N. A. M., Warid, M. N. M., Satar, M. K. I. M., and Mashros, N. (2018, April). Effect of fast pyrolysis bio-oil from palm oil empty fruit bunch on bitumen properties. In *IOP Conference Series: Materials Science and Engineering* (Vol. 342, No. 1, p. 012053). IOP Publishing.
- (b) **Raman, N. A. A.**, Hainin, M. R., Hassan, N. A., and Ani, F. N. (2015). A review on the application of bio-oil as an additive for asphalt. *Jurnal Teknologi (Sciences and Engineering)*, 72(5), 105–110.
- (c) **Raman, N. A.A.**, Hainin, M. R., Hassan, N.A, Ani, F. N., Warid, M.N.M., Satar, M. K. I. M., and Mamat, R. (2015). Effect of bio-oil from empty fruit bunch on penetration index of asphalt binder. *Jurnal Teknologi (Sciences and Engineering)*, 77(23), 13-6.