BINDER CHARACTERIZATION AND PERFORMANCE OF ASPHALTIC CONCRETE MODIFIED WITH WASTE COOKING OIL

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“To my beloved parents, siblings and friends who are always behind and supporting me”

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ABSTRACT

The use of waste cooking oil (WCO) in binder modification is widely explored in response to waste management issue. However, the decreasing rheological performance pattern trend at high temperature by using WCO is globally recorded and yet still unresolved. This poor performance is due to the high acidity of the WCO. To resolve this issue, a chemical treatment was proposed to reduce the acidity of the WCO. Therefore, the aim of this study was to evaluate the performance of binders modified with untreated and treated WCO. It was carried out in three phases. In Phase 1, the physical and rheological tests of binder (penetration, softening point, viscosity and DSR) were conducted to determine the optimum percentages of untreated and treated WCO (between 0, 5, 10, 15 and 20%) in modifying the binder. In Phase 2, the optimums WCOs were utilised for further mechanical performance evaluation of Asphaltic Concrete 14 (AC14) mixture through Marshall stability, resilient modulus, dynamic creep and indirect tensile strength (ITS). The morphology and microstructure observations were performed in Phase 3 to investigate the adhesion bonding between modified binder and aggregates. The test results showed that the acidity of the WCO decreased after chemical treatment. The rheological test showed that the failure temperature of binder modified using the treated WCO has increased to 70 ^{o}C. In addition, treated WCO mixture recorded superior performance by being less susceptible to permanent deformation as compared to the control mix. Besides, the microstructure analysis revealed that low surface roughness of binder modified with treated WCO has strengthened the adhesion bonding with aggregates. In conclusion, the chemical treatment had improved the treated WCO performance in the modified binder as asphalt paving materials.

ABSTRAK

Penggunaan sisa minyak masak (WCO) dalam pengubahsuaian pengikat diterokai secara meluas sebagai respon kepada isu pengurusan sisa. Namun, penurunan pola corak prestasi reologi pada suhu tinggi dengan menggunakan WCO telah direkodkan secara global dan masih belum diselesaikan lagi. Prestasi lemah ini disebabkan oleh keasidan WCO yang tinggi. Untuk menyelesaikan isu ini, rawatan kimia dicadangkan untuk mengurangkan keasidan WCO. Oleh itu, matlamat kajian ini adalah untuk menilai prestasi pengikat diubahsuai dengan WCO yang tidak dirawat dan dirawat. Ianya dijalankan dalam tiga fasa. Fasa pertama, ujian fizikal dan reologi untuk pengikat (penusukan, titik lembut, kelikatan dan reometer ricih dinamik) dijalankan untuk menentukan peratusan optimum WCO yang tidak dirawat dan dirawat (antara 0, 5, 10, 15 dan 20%) dalam mengubahsuai pengikat. Fasa kedua, optimum WCO digunakan untuk penilaian prestasi mekanikal campuran asfalt konkrit 14 (AC14) melalui kestabilan Marshall, modulus kebingkasan, rayapan dinamik dan kekuatan tegangan tidak langsung (ITS). Pemerhatian morfologi dan mikrostruktur dilakukan dalam fasa ketiga untuk menyiasat ikatan lekatan antara pengikat diubahsuai dan agregat. Keputusan ujian menunjukkan keasidan WCO menurun selepas rawatan kimia. Ujian reologi menunjukkan suhu kegagalan untuk pengikat diubahsuai menggunakan WCO dirawat telah meningkat kepada 70 ^{o}C. Tambahan, campuran WCO dirawat merekodkan prestasi lebih baik yang kurang terdedah kepada ubah bentuk kekal berbanding campuran kawalan. Selain itu, analisis mikrostruktur mendedahkan kekasaran permukaan rendah oleh pengikat diubahsuai dengan WCO dirawat memperkuatkan ikatan lekatan dengan agregat. Kesimpulannya, rawatan kimia meningkatkan prestasi WCO dirawat dalam pengikat diubahsuai sebagai bahan turapan asfalt.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AC 14	-	Asphaltic concrete of nominal maximum aggregate size 14 mm
AFM	-	Atomic force microscopy
AI	-	Aging index
Al	-	Aluminum
ANOVA	-	Analysis of Variance
ASTM	-	American Society for Testing Materials
BBR	-	Bending beam rheometer
COD	-	Chemical oxygen demand
CSED	-	Critical strain energy density
DSR	-	Dynamic shear rheometer
EDX	-	Energy dispersive x-ray spectroscopy
Fe	-	Iron
FESEM	-	Field emission scanning electron microscope
FFA	-	Free fatty acid
FTIR	-	Fourier transform infrared
GCMS	-	Gas chromatography-mass spectrometry
HMA	-	Hot Mix Asphalt
ITS	-	Indirect tensile strength
ITSM	-	Indirect tensile stiffness modulus
JKR	-	Jabatan Kerja Raya
K	-	Potassium
KOH	-	Potassium hydroxide
LVDT	-	Linear Variable Displacement Transducer
MSCR	-	Multiple stress creep and recovery
NaOH	-	Sodium hydroxide

OBC - Optimum bitumen content
PVN - Penetration viscosity number
RTFO - Rolling thin film oven
TMD - Theoretical maximum density
USD - Universal sorption device
UTM - Universiti Teknologi Malaysia
UVO - Used vegetable oil
VFB - Voids filled with bitumen
VTM - Voids in total mix
WCO - Waste cooking oil
WMA - Warm mix asphalt
WVO - Waste vegetable oil
XRD - X-ray diffraction

LIST OF SYMBOLS

c - y-intercept
^{o}C - Celsius
cm - centimetre
cm^{-1} - reciprocal wavelength
cm^{3} - cubic centimetre
cP - centiPoise
cSt - centiStokes
dmm - decimillimetre
^{o}F - Fahrenheit
g - gram
G* - complex modulus
h - hour
Hz - Hertz
J - Joules
J_{nr} - Nonrecoverable creep compliance
kg - kilogram
km - kilometre
kPa - kilo Pascal
kv - kilovolt
m - slope
m^{3} - cubic metre
meq - miliequivalents
mg - milligram
ml - millilitre
mm - millimetre

mm^2 - millimetre square

μm - micrometre

MPa - Mega Pascal

M_R - Resilient modulus

N - Newton

N KOH - Molarity KOH

nm - nanometre

Pa.s - Pascal seconds

R^2 - Regression

rpm - revolution per minute

s - second

% - percentage

δ - Delta/phase angle

σ - applied stress

ε - recoverable strain

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Petroleum-based asphalt binder is derived from petroleum refinement process by-product (Wen *et al.*, 2013). Bitumen is recognised as an essential coating material in bituminous pavements composition apart from the aggregates skeleton for pavement construction. In asphalt mixture composition, the binder functions as an adhesive agent for coating process and binds the aggregate particles together. Generally, pure bitumen production through crude oil petroleum refining process is not desirable in road pavement application. The asphalt binder exhibits insufficient properties for pavement construction and need to be modified with various additives types such as carbonaceous materials, fine minerals and polymers (Chebil *et al.*, 2000). An improvement of engineering properties for asphalt binder can be achieved with the application of a modifier by reducing temperature susceptibility and enhancing the rheological performance to withstand the environmental and traffic loading. Currently, a number of notable studies are being conducted worldwide to explore valuable resources from waste materials as a modifier for asphalt binder modification.

In recent years, a wide range of oil-based modifications have been introduced, especially involving waste cooking oil (WCO). The WCO is also recognised as waste grease oil which is characterised as the by-product of fresh cooking oil produced during cooking and food processing. This oil source has recently gained widespread attention because of its satisfactory achievement as a potential waste material to enhance the physical and rheological performance of modified binder. The oil undergoes three types of common chemical reaction during frying, such as hydrolysis, oxidation and polymerisation. The chemical process in oil causes degradation in the physical and chemical properties (Cvengroš and Cvengrošová, 2004) which affects WCO quality. Physical properties include alteration in foaming quantity, colour, viscosity, density and flavour. Meanwhile chemical properties are represented as total unsaturation compound, free fatty acid content, polar and polymeric material. The generation of huge quantities of WCO is attributed by the increasing human population and its use as a frequent medium for food preparation involving frying. It can be noticed that WCO production quantity is directly proportional to frying rate.

According to Chhetri *et al.* (2008), enormous quantity of WCO that is generated worldwide is illegally dumped and released into the surrounding environment. In Malaysia, the disposal of WCO is reported to be approximately around 50,000 tons, which was produced from plant and animal based fats source (Yaakob *et al.*, 2013). These wastes are disposed to the environment without undergoing any proper treatment (Kheang *et al.*, 2006). A survey conducted by Kabir *et al.* (2014) revealed that the majority of the respondents (54.5%) discard WCO into their house sink. Meanwhile 22.2% stated that the WCO is dumped into drains. This coincides with the response on the level of awareness on WCO recycling. Unexpectedly, only 12% of the households WCO is recycled, while most of the respondents (about 88%) did not practice waste recycling as they discharge WCO improperly. Such inappropriate action has consequently induced an undesirable impact to the entire environmental ecosystem, for instance distraction of aquatic life, contamination of water and soil, sewer system blockage and increased maintenance cost for water treatment and waste management (Chen *et al.*, 2009).

Abundant of WCO production can cause prominent adverse impact and threat to the environment if not properly managed and disposed. Therefore, recycling or reusing WCO in modified asphalt binder is considered as an effective utilisation and management of this waste while at the same time ensuring economic and environmental benefits (Patil *et al.*, 2012). It is noteworthy that, most researchers have focused on the superior performance of WCO as a rejuvenator for aged binder (Asli and Karim, 2011; Zargar *et al.*, 2012; Asli *et al.*, 2012; Zaumanis *et al.*, 2013; Chen *et al.*, 2014a; Binbin *et al.*, 2014; Chen *et al.*, 2014b), apart from substituting WCO in modified binder to improve rheological performance. The WCO performance as a modifier at high and low temperatures was evaluated by Wen *et al.* (2013). The rheological findings indicate declination of the complex modulus (G*), which resulted in a low rutting resistance at high temperature. On the contrary, an increment in thermal cracking resistance performance at low-temperature was observed to occur linearly with the addition of WCO content.

This rheological result coincides with the study conducted by Maharaj *et al.* (2015) for un-aged sample, wherein an enhancement of fatigue cracking resistance was achieved at low temperature. Meanwhile, the high temperature performance showed an adverse effect as the rutting resistance decreased with the addition of WCO. This is also supported by Sun *et al.* (2016), which reveals the decrement of deformation resistance and improvement of thermal cracking resistance performance when using the modified binder incorporated with bio-oil derived from WCO. According to Teymourpour *et al.* (2015), the application of WCO in conventional binder proved an enhancement of thermal cracking resistance but compromising the high temperature performance as evidenced by the reduction of resistance to rutting. The superiority performance of fatigue cracking resistance was noticeable with the addition of WCO as reported in a previous research. It proves that the capability of WCO to improve low temperature performance, while deteriorating the rutting resistance performance at high temperature.

The WCO in asphalt binder softens the physical properties of modified asphalt binder, thereby unable to withstand rutting exposure. According to Katamine

(2000), the negative indicator in the quality of bitumen is expected with the addition of WCO in bitumen. This serves as the major reason why WCO is not suggested for optimal utilisation, due to its poor performance in rutting resistance, which is not desirable especially for hot climatic region. Owing to the adverse and unsatisfactory performance in rheological properties, no further research work was conducted to evaluate the mechanical properties of asphalt mixture. This is due to the expectation of high tendency for rutting problem, thereby reducing the strength of asphalt mixture. Waste oil needs to be fully re-evaluated and assessed by undergoing further treatment before being recommended and applied in the asphalt binder to produce bituminous mixture (Borhan *et al.*, 2009).

However, despite the broadly practiced WCO application, the identification of fundamental parameters influencing WCO in binder modification and also the effectiveness of the process has not been investigated yet (Teymourpour *et al.*, 2015). In addition, the mechanism for modifying asphalt binder with WCO and the effect of WCO on the modified binder performance are not clearly documented. Obviously, the softer and less viscous modified binder with WCO depicts the weakness of internal chemical bonding of the material. Hence, there is the issue of chemical compatibility and interprets an assumption of incompatibility between these two materials due to chemical properties that should be further clarified and investigated.

1.2 Problem Statement

A high amount of WCO is generated due to abundant fresh cooking oil consumption during frying. Inappropriate waste disposal and dumping into the landfill has induced an adverse impact to the surrounding environmental ecosystem. Therefore, a waste management strategy arises to mitigate environmental pollution due to the large quantity of WCO. Apart from waste disposal, waste utilisation has become a valuable option due to the realisation on the significance of awareness level for environmental preservation. Essentially, it is highly recommended to adopt a more sustainable approach of recycling WCO product in modified asphalt binder as a pavement material in road construction. The application of WCO in asphalt binder indicates an effective solution of waste management which is gaining extensive interest from other researchers.

Most of the previous studies have concentrated on the modified binder incorporated with WCO (Wen *et al.*, 2013; Maharaj *et al.*, 2015). However, none of the researchers emphasised on the basic parameter affecting oil modification (Teymourpour *et al.*, 2015). In addition, this parameter is not clearly explained since the modification of asphalt binder with WCO is still in the empirical stage. Yet, WCO is directly used in modified asphalt binder without determining the quality of WCO. Theoretically, with regard to the modified binder performance, the rheological evaluation is affected by the quality of WCO, in which the acid value is represented as one of the main parameters for quality measurement specifically. Preliminary finding has revealed that an increased acid value has caused the decreasing pattern trend in rheological performance and vice versa. The increment in acid value is attributed to the degradation rate of WCO during frying and exhibits undesirable characteristic that constraint the optimisation of WCO in asphalt binder. Therefore, there is an urgency for WCO to undergo a chemical pre-treatment in minimising the existing high acid value before being utilised for the modification of asphalt binder. It is noteworthy to compare the performance of modified binder containing untreated and treated WCO, and identify any rheological and mechanical performance improvement after conducting the chemical modification.

In addition, the softer and less viscous modified binder with WCO is noticeable and hinders the application of WCO in high temperature region due to high rutting exposure. These poor properties depict the weakness in the internal chemical bonding and portray the disturbance of molecule particle structure of the material. It gives an assumption that there is a restrictive factor influencing the refusal of molecule interaction to form strong chemical bonding in the modified asphalt binder with WCO. Therefore, the issue of chemical compatibility between

WCO and asphalt binder arises and should be investigated (Gong *et al.*, 2016). Since there is no previous research conducted to investigate the compatibility issue, there is a need to conduct a chemical analysis in order to characterise the chemical compatibility properties between these two materials in this study. This factor contributes to the main reason as to why the level of WCO usage is comparatively very low, and implies that there is a requirement to evaluate the suitability of WCO as a modifier, which is available at low cost feedstock without compromising the quality performance of modified binder with WCO as paving materials.

1.3 Aim and Objectives of the Study

This study aims to evaluate an asphalt mixture incorporating untreated and treated WCO in modified binder. In order to achieve this aim, the study is carried out to fulfil the following objectives:

1. To determine the properties of untreated and treated WCO in modified binder for physical and rheological performance.
2. To evaluate the mechanical performance of Hot Mix Asphalt (HMA) incorporating untreated and treated WCO.
3. To analyse the morphology and microstructure characteristics of modified binder and asphalt mix containing untreated and treated WCO.

1.4 Scope of the Work

The scope of this research is to enhance binder properties with the upgraded of WCO quality as a modifier through chemical treatment. The improved mechanical performance exhibited by asphalt mixture incorporating treated WCO is expected to be more superior and comparable to the conventional asphalt mixtures. Raw WCO derived from palm oil was collected from a café surrounding UTM, Skudai. The aggregates sample source was obtained from Hanson Quarry in Kulai, Johor. The control asphalt binder used in this study was PEN 60/70 which was sourced from Chevron Company and has met all the specifications as outlined by the Jabatan Kerja Raya (2008) specifications.

The basic test for binder evaluation was performed which included penetration test, softening point test, viscosity and dynamic shear rheometer (DSR). The modified asphalt binder containing untreated and treated WCO underwent an aging condition by conducting a rolling thin film oven (RTFO) only, which is restricted for the assessment of rutting resistance at high temperature, and does not address the fatigue cracking performance. This implies that this research is focused on the rutting resistance performance since poor performance was recorded by the previous work that should be improved in this study.

An optimum WCO percentage was selected based on binder evaluation for further mechanical performance testing of asphalt mixture. The asphalt mixtures were evaluated based on the Marshall Stability and flow test, resilient modulus test, dynamic creep test and indirect tensile strength test. The control mixture, untreated WCO mixture and treated WCO mixture, were compared in order to identify any mechanical performance improvement. The microstructure observation was conducted by using Atomic Force Microscopy (AFM), Gas Chromatography-Mass Spectrometry (GCMS), Fourier Transform Infrared (FTIR), Field Emission Scanning Electron Microscope (FESEM) and X-Ray Diffraction (XRD). Most of the chemical treatment of WCO, asphalt binder and bituminous mixtures testing and microstructure analysis were performed at the Chemical Laboratory, Transportation and Highway Laboratory and Central Laboratory at UTM Skudai, Johor.

1.5 Significance of Study

WCO is identified as a valuable potential waste material that has capability to enhance the performance of conventional asphalt binder for road pavement construction. The superior physical and chemical properties exhibited by WCO source attribute to the selection of this waste to be applied as a modifier in the modification of asphalt binder. WCO can give adverse impact if not properly managed and disposed. On the contrary, an effective management of this waste, by recycling WCO in modified asphalt binder for paving materials, significantly contributes to the advantages in three principles, such as environmental, economic, and social.

From an environmental stand point, the utilisation of abundant WCO by recycling this waste into paving materials is seen as an efficient sustainable effort which minimises the generation of waste dumping in the landfill and illegal waste disposal into the drainage system, reduces the consumption of natural resource and preserves the environmental ecosystem as well. When WCO is discharged into the river, the formation of a thin oil layer on the water surface attributes to the blockage of sunlight energy source to pass through, that is required by the marine environment. In addition, the presence of WCO in the water system causes the alteration of oxygenation process by disrupting oxygen supply to the aqua marine ecosystem. The increment of chemical oxygen demand (COD) due to WCO existence causes water contamination in the presence of poisonous compound thus destroying aquatic life. Meanwhile, for residential area, WCO accumulation has induced drainage system blockage thus causing clogging problem, which in turn increases the maintenance cost to fix and monitor this issue. Obviously, by recycling WCO, it can mitigate and solve the environmental problem directly.

From an economical point of view, the cost of road construction may be minimised by the usage of WCO as modifier. WCO is selected because of its low-cost feedstock to be obtained and available abundantly (Villanueva *et al.*, 2008).

Despite the cheap price, the upgraded WCO properties by chemical modification exhibit better rheological and mechanical performance for asphalt mixture. In comparison to other highly cost modifiers, WCO source has the capability to deliver high performance potential as compared to other modifiers. Sustained utilisation of WCO as a modifier in binder will be able to drastically reduce the waste disposal of enormous WCO quantity.

The recyclability of WCO for paving material in pavement construction industry has proved an attempt to raise awareness among Malaysian in increasing the WCO management practice, thus improving the quality of social life based on the social standpoint. However, this practice is still at an initial stage. Therefore, it is necessary to empower public awareness campaign to encourage household toward proper WCO management. The awareness campaign should briefly cover the negative impact of inappropriate disposal of WCO and emphasise the significance of WCO recycling for environmental benefits as well. In implementing this valuable awareness, support from the government is required to ensure successful WCO recycling programmes. The government should encourage citizens and industries through advertisement and provide additional information that would be helpful in improving the WCO recycling management.

1.6 Summary

The deficiency of the rutting resistance performance exhibited by modified binder and asphalt mixture when dealing with oil-based source can be solved by the improvement of untreated WCO properties through chemical modification. A comparison on the performance of binder and asphalt mix incorporating untreated and treated WCO, which has not been previously studied, is explored in this research. In this regard, the study consists of three types of evaluation in terms of binder (penetration, softening point, viscosity and DSR), mechanical asphalt mixture

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