



UNIVERSITI PUTRA MALAYSIA

***AGING DEFORMATION RESISTANCE OF NANOCLAY-
MODIFIED STONE MASTIC ASPHALT MIXTURE***

LAMYA M. J. MAHDI

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**AGING DEFORMATION RESISTANCE OF NANOCCLAY-
MODIFIED STONE MASTIC ASPHALT MIXTURE**

By

LAMYA M. J. MAHDI

**Thesis Submitted to the School of Graduate Studies, Universiti
Putra Malaysia, in Fulfillment of the Requirement for the
Degree of Doctor of Philosophy**

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DEDICATION

Dedicated to my beloved husband Dr. Ali Radi Mahdi, who gave me a chance to figure out myself and being with me hand in hand, I will always owe him for his love, support and patience and for sacrificing all so that I can achieve my Ph.D.

To my beloved mother, sisters: Dr. Aida, Dr. Thana, Engineer Alaa and Methal and brothers: Jamal and Engineer Mahdi for their love, and encouragements towards the success of this study.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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March 2015

Chairman: Professor Ratnasamy Muniandy, PhD
Faculty: Engineering

Many modifiers have been used to modify bitumen such as fiber, polymer, and filler which cost the road authorities a lot of money in addition to their side effects on the environment and their limited advantage. Aging have limited the service life of pavement and accelerated its premature failures. The problem of asphalt aging still exists in spite of the extensive use of the above mentioned modifiers.

Montmorillonite nanoclay is one of the hybrid materials having a favored layer structure distinguish it from other materials in ability to disperse on a nanometer scale. It is a natural source material with a relatively low cost and more importantly environmentally friendly. The successfully intercalation chemistry of the layered silicates with polymers has been widely studied for a long time, and properties such as thermal, mechanical showed superior enhancement.

Based on these findings, this study presents a laboratory investigation into the performance of asphalt binder and Stone Mastic Asphalt mixture modified with different type and concentration of nanoclay.

The Rolling Thin Film Oven method was used to simulate the short term aging of the base and nanoclay modified asphalt binder, while the Stone Mastic Asphalt mixture was aged according to the asphalt mixture specification. The Dynamic Shear Rheometer was performed to characterize the rheological properties of asphalt binder at different temperatures and frequencies. The effect of nanoclay on the performance of the Stone Mastic Asphalt mixture was also investigated by means of the Indirect Tension Test and the Repeated Load Axial Test.

The microscopic images indicated a homogenous dispersion of the layered silicate in asphalt medium while the spectroscopic technique showed the incorporation of the nanoclay in the asphalt binder matrix via new functional groups. Nanoclay was found to have a significant influence on asphalt binder and mixture properties. The higher nanoclay concentration showed better performance. The asphalt binder physical, rheological properties and rutting resistance were positively improved after the addition of nanoclay.

The permanent deformation resistance of the Stone Mastic Asphalt mixture was found to be enhanced due to the stiffening effect of the nanoclay. Meanwhile, inconsistent trend was observed among the concentrations and types of nanoclay especially with permanent deformation test results, however, both of nanoclay types showed remarkable improvement as compared with the control mixture. The reason can be contributed to the complicated asphalt mixture failure mechanism since different variables having heterogeneous characters contributed in construction the Stone Mastic Asphalt mixture while using very close nanoclay concentration from each others can be a another reason.

The short term aging resistance of modified binder was found to be improved which indicated the ability of layered nanoclay to act as a barrier that restricted and delayed the penetration of oxygen and played an important role to reduce the volatilization of the bitumen's oily components and as consequence improved the asphalt binder resistance to rutting while enhanced the engineering properties of SMA mixtures as providing resistance against permanent deformation.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

KESAN PENUAAN BAGI RINTANGAN BAGI NANOCLAY CAMPURAN STONE MASTIC ASPHALT YANG DIUBAHSUAI

Oleh

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Mac 2015

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Kebanyakan pengubah telah digunakan untuk mengubah suai bitumen seperti serat, polimer dan pengisi yang menyebabkan pihak berkuasa jalan raya menelan belanja yang banyak di samping kesan sampingan terhadap alam sekitar dan kelebihan yang terhad. Penuaan telah menghadkan jangka hayat perkhidmatan turapan dan mempercepatkan kegagalan pra-matang itu. Masalah penuaan asfalt masih wujud walaupun penggunaan meluas di atas pengubahsuaian tambahan.

Montmorilonit nanoclay adalah salah satu bahan hibrid yang mempunyai struktur lapisan yang disukai membezakannya daripada bahan-bahan lain dalam keupayaan untuk bersurai pada skala nanometer. Ia adalah bahan sumber semulajadi dengan kos yang agak rendah dan lebih penting lagi mesra alam. Kimia interkalasi telah berjaya daripada silikat berlapis dengan polimer telah dikaji secara meluas untuk jangka masa yang lama, dan sifat-sifat seperti haba, mekanikal menunjukkan peningkatan unggul.

Berdasarkan hasil dapatan, kajian ini membentangkan satu kajian makmal ke dalam prestasi pengikat asfalt dan campuran Batuan Mastik Asfalt diubahsuai dengan jenis yang berbeza dan konsentrasi nanoclay.

Kaedah Rolling Thin Film Oven telah digunakan untuk mensimulasikan jangka pendek penuaan daripada asas dan nanoclay diubahsuai asfalt pengikat, manakala campuran Batuan Mastik Asfalt telah berumur mengikut spesifikasi campuran asfalt. Dinamik Ricih Reometer telah dijalankan untuk mencirikan sifat-sifat reologi pengikat asfalt pada suhu yang berbeza dan frekuensi. Kesan daripada

nanoclay kepada prestasi campuran Batuan Mastik Asfalt juga disiasat melalui Ujian Ketegangan Tidak Langsung dan Beban Paksi Berulang Ujian.

Imej-imej mikroskopik menunjukkan penyebaran homogen daripada silikat yang berlapis dalam medium asfalt manakala teknik spektroskopi yang menunjukkan pemerbadanan nanoclay dalam matriks pengikat asfalt melalui kumpulan berfungsi baru. Nanoclay didapati mempunyai pengaruh yang besar ke atas pengikat asfalt dan sifat-sifat campuran. Kepekatan nanoclay yang lebih tinggi menunjukkan prestasi yang lebih baik. Asfalt pengikat fizikal, sifat reologi dan rintangan aluran telah positif bertambah baik setelah penambahan nanoclay.

Ketahanan ubah bentuk kekal daripada campuran Batuan Mastik Asfalt didapati untuk mempertingkatkan oleh pengerasan kesan daripada nanoclay. Sementara itu, trend yang tidak konsisten diperhatikan dalam kalangan kepekatan dan jenis nanoclay terutamanya dengan keputusan ujian ubah bentuk kekal, bagaimanapun, kedua-dua jenis nanoclay menunjukkan peningkatan yang memberangsangkan berbanding dengan campuran kawalan. Alasan boleh menyumbang kepada mekanisme kegagalan campuran asfalt rumit sejak pembolehubah yang berbeza mempunyai heterogen ciri-ciri yang menyumbang dalam pembinaan campuran Batuan Mastik Asfalt semasa menggunakan nanoclay kepekatan yang sangat rapat antara satu sama lain boleh sebab yang lain.

Penuaan rintangan jangka pendek daripada diubahsuai pengikat telah didapati bertambah baik yang menunjukkan keupayaan nanoclay berlapis untuk bertindak sebagai penghalang yang menyekat dan melambatkan penembusan oksigen dan memainkan peranan penting untuk mengurangkan pemeruapan komponen berminyak bitumen dan mengakibatkan peningkatan asfalt rintangan pengikat untuk aluran dan meningkatkan sifat-sifat kejuruteraan campuran Batuan Mastik Asfalt menyediakan rintangan terhadap ubah bentuk kekal.

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Last but not least, I would like to express my gratitude to all my family: mother, brothers and sisters, for their love and encouragement.

I certify that a Thesis Examination Committee has met on 31 March 2015 to conduct the final examination of Lamya M. J. Mahdi on her thesis entitled "Aging Deformation Resistance of Nanoclay-Modified Stone Mastic Asphalt Mixture" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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LIST OF ABBREVIATIONS

AASHTO	American Association of State Highway and Transportation Officials
ANOVA	Analysis of Variance
ASTM	American Society for Testing and Materials
BET	Brunauer Emmett Teller
BS	British Standard
BT	Bentonite Clay
CAPA	Colorado Asphalt Pavement Association
CEC	Cation Exchange Capacity
COPF	Cellulose Oil Palm Fiber
DLS	Dynamic Light Scattering
DSR	Dynamic Shear Rheometer
EPDM	Ethylene Propylene Diene Monomer
EVA	Ethyl Vinyl Acetate
FHWA	Federal Highway Administration
FN	Flow Number
FTIR	Fourier Transform Infrared Spectroscopy
GEL	Gelatinous
H/C	Hydrogen to Carbon Ratio
HMA	Hot Mix Asphalt
Hz	Hertz
Ic	Index of Colloidal Instability
ISP	Increment in Softening Point
ITT	Indirect Tension Test
KC	Kaolinite Clay
KRIP	Kajang Rocks Innopave Premix Company
LA	Los Angeles Abrasion
LL	Lower Limit
LVDT	Linear Variable Differential Transducer
LVE	Linear Viscoelastic
MATTA	Material Testing Apparatus
MMT	Montmorillonite
MS	Measurement Systems
Na-MMT	Sodium Montmorillonite
NAPA	National Asphalt Pavement Association
NCHRP	National Cooperative Highway Research Program
NMAS	Nominal Maximum Aggregate Size

OAC	Optimum Asphalt Content
OBT	Organic Modified Bentonite
OMMT	Organic Montmorillonite
OSU	Oregon State University
PAV	Pressure Aging Vessel
PG	Performance Grade
PI	Penetration Index
PVC	Polyvinyl Chloride
R&B	Ring and Ball
RLAT	Repeated Load Axial Test
RMP	Refinery Mechanical Pulping
RP	Retained Penetration
RPM	Revolutions per Minute
RTFO	Rolling Thin Film Oven
RV	Rotational Viscometer
SABITA	Southern African Bitumen Association
SBR	Styrene Butadiene Rubber
SBS	Styrene Butadiene Styrene
SHRP	Strategic Highway Research Program
SIS	Styrene Isoprene Styrene
SMA	Stone Matrix Asphalt
SOL	Solution
SSD	Saturated Surface Dry
STA	Short Term Aging
SUPERPAVE	Superior Performing Asphalt Pavement
TEM	Transmission Electron Microscopy
TMD	Theoretical Maximum Density
TR	Crumb Rubber
TSR	Tensile Strength Ratio
UKM	Universiti Kebangsaan Malaysia
UL	Upper Limit
UPM	Universiti Putra Malaysia
UTM	Universal Testing Machine
UV	Ultraviolet
VAI	Viscosity Aging Index
VCA	Voids in Coarse Aggregate
VFA	Voids filled with Asphalt
VMA	Voids in Mineral Aggregates
VTM	Voids in Total Mix
VTS	Viscosity Temperature Susceptibility

Wc	Work Dissipated per Loading Cycle
WDXRF	Wavelength Dispersive X-Ray Fluorescence
WPE	Waste Packaging Polyethylene
WSDOT	Washington State Department of Transportation
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

CHAPTER 1

INTRODUCTION

1.1 Background

The nanotechnology research has attracted great interest from researcher throughout the world due to the various interesting functions of materials on nanoscale dimensions and having relatively larger surface-area-to-mass ratio which make them more chemically reactive and positively change their strength or other important properties and thus increase their potential to use in a wide range of applications.

Nanoscale materials which is a material having at least one dimension sized from 1 to 100 nanometers, have been adopted by many of the industries and academics and showed unexpected reaction by the community, announcing a new industrial revolution. One of the most attractive nanomaterials is nanoclay which is a natural occurring material widely used by researcher from long time because of its availability, relatively low cost, environmentally friendly and the most important property which is its favorable and unique structure make it more compatible to create nanocomposite. Montmorillonite nanoclay which is the most commonly used layered silicates has been successfully used to reinforce polymers (Ray and Okamoto, 2003). Montmorillonite nanoclay is anisotropy filler which has large aspect ratio (length to thickness ratio) make it especially favorable in matrix reinforcement (Patel et al., 2006).

Montmorillonite from smectite group clays has a 2:1 phyllosilicates layer structure which composed of a shared octahedral sheet sandwiched between two tetrahedral sheets (Okada and Usuki, 2006). In order to provide more compatible nanocomposite with various polymer types, montmorillonite is treated with special types of surfactants such as quaternary ammonium salt through an ion exchange reaction (Giannelis, 1996). Many of polymer properties can successfully enhanced with the addition of few amount of nanoclay (Alexandre and Dubois, 2000).

Recently nanoclay attracted the attention of the asphalt pavement researchers and with the expected of more significant result, small amount of nanoclay was added as a third part to enhance the compatibility between the polymer and asphalt binder, the results showed remarkable improvement in modified binder's properties comparing with the unmodified one (Yu et al., 2007; Polacco et al., 2008; Zhang et al., 2009; Sureshkumar et al., 2010; Galooyak et al., 2010).

The positive results that gained from adding nanoclay to polymer asphalt nanocomposite gave the pavement researchers motivation to adopt this material which enables them to study the asphalt binder behavior at nano scale level.

A lot of studies have been done on nanoclay modified polymers and other materials but the research studies on this promising material to modified asphalt binder is still unclear due to lack of published research on this aspect. Also, very little information is available regarding the effect of nano materials on the characteristics, rheological properties, and viscoelastic behavior of nano material modified asphalt (nanocomposite) and very rare research concerning the effect of nanoclay on asphalt mixture. However, the characteristics of this material can significantly influence the properties of the nanocomposite, and thus can have a significant effect on mixture performance. To better understand and characterize the interaction of nanoclay with the asphalt binder, more effort and studies need to be carried out on nanoclay modified asphalt binder to determine its performance. It is believed that the focus on the asphalt binder rheological properties can have a significant effect in the practical application of nanoclay modified asphalt. Thus, beside the physical properties, this study will focus on the investigation of rheological and deformation properties of unaged and aged bituminous binders and Stone Mastic Asphalt (SMA) mixture, which is considered to be an effective and common approach to obtain the desired purpose mentioned above.

The type and quantity of nanoclay used to modify bitumen are especially important in particularly mixes like Stone Mastic Asphalt (SMA) mixtures where the properties of the mortar consisting of bitumen, fine aggregates, filler, and fiber and acts as a real glue in the asphalt mixture to stabilize the stone aggregate skeleton in an asphalt pavement and thus contributes significantly to compatibility impermeability, adhesion, and in-service pavement performance. Nanoclay varies depending on their groups, sources and the type of surfactant that used to modify them; as a consequence each nanoclay has different performance from others when they use as the reinforcement phase in various matrices.

1.2 Problem Statement

One of the most important issues facing asphalt pavement researchers as a consequence to the increasing traffic loads, traffic volumes, number of vehicle axle load, and environmental factors, is the kind of modifier that need it to improve the properties of the existing bituminous material to ensure provide longer service life, less maintenance, cheaper, and environmental friendly asphalt pavements. During its service life, asphalt pavement experience premature failures such as fatigue, rutting, thermal cracking, and moisture induced damage, this lead to urgent resolution to limit or at least delay this kind of failure which cost the road authority a lot of money with the ever increasing of crude oil prices.

On the other hand, due to the high-temperature, aging has been limited the service life of pavement. The problem of asphalt aging still exists in spite of the extensive

use of modification additives like polymer, filler and fiber in addition of their high cost and possible environmental concerns. At high service temperatures, and relatively low frequency of loading, bitumen because of its viscoelastic properties can become soft with a low stiffness and more susceptible to permanent deformation. Bitumen can easily get aged when exposure to elevated temperature and air due to the diffusion of oxygen and UV radiation.

Due to the relatively new technology introduced on pavement sector and the lack of the published research concerning this aspect; the stiffness and deformation resistance of the asphalt binder modified with nanoclay needs to be explored to provide insight into the performance of asphalt mixture. Based on the promising characteristics of the nanoclay that significantly influence the properties of the asphalt binder (Ghile, 2006), as a consequence their effect on the mixture performance need to be investigated.

The performance of nanoclay in asphalt mixture is unclear due to the few researches available concerning the interaction effects of nanoclay with asphalt mixture. The importance of nanoclay has been more over looked in the asphalt binder than mixture.

1.3 Objectives of the Study

The primary aim of this research is to investigate the possibility of using montmorillonite nanoclay with different types and concentrations to enhance the characteristics of the base bitumen and consequently to examine their potential effect on Stone Mastic Asphalt (SMA) mixtures performance. In order to achieve these aims, the following objectives were defined:

1. To evaluate the effects of nanoclay type and content on physical and rheological properties of asphalt binder before and after aging process.
2. To determine the effect of nanoclay modified binder on Stone Mastic Asphalt (SMA) mixture in terms of their permanent deformation properties before and after aging process.

1.4 Significance of the Study

The knowledge produced by this research can give contribution to develop a superior performing pavements by introducing new binder has the characteristics of lower temperature susceptibility and aging effect and thus can mitigate high temperature rutting throughout naturally occurring material that can reduce the cost of highway construction and maintenance as well as contribute to resolve one of the environmental problems. Nanoclay in this study tends to be economical, environmentally sound and effective to improve the pavement performance.

In addition, results of this research will contribute to a better characterize the interaction of nanoclay with the bitumen and give insight to their significant effects on the performance properties of binder and particularly Stone Mastic Asphalt (SMA) mixtures.

Furthermore, results of this study will give insight about the significance of the use of nanoclay in asphalt pavements particularly SMA mixtures to prevent or at least delay the premature pavement failure and consequently save life cycle cost. Results of this study will enable road authority to make proper judgment regarding the use of nanoclay in asphalt mixtures as a consequence can save a lot of money on maintenance and rehabilitation work of the roads.

1.5 Scope and Limitation

The main goal of this research is to investigate the effect of different montmorillonite nanoclay types and concentrations on the physical and rheological properties and viscoelastic behavior of asphalt binder and consequently on the performance of Stone Mastic Asphalt (SMA) mixture. This research test results are limited best on the following:

- x Only two types of montmorillonite nanoclay were used in this study which prepared from the same origin montmorillonite clay but modified using two different types of surfactant in order to convert it to organic montmorillonite.
- x This study focused only on the effects of short term aging and how it performs on asphalt binder and Stone Mastic Asphalt (SMA) mixture.

1.6 Thesis Layout

According to the objectives, the layout of the thesis is as follows:

Chapter 1 presents the introduction, research problem, objectives, significance of the study, and scope and limitation. Chapter 2 elaborates the literature review concerning subjects related to the structure, properties, and applications of nanoclay as well as bitumen origin, application, constitution, structure, and properties. Other works related to the nanoclay modified asphalt binder and mixture. In addition to the aging process of both modified asphalt binder and mixture. Description of DSR procedure and test performed. SMA Mix design and failure mechanisms were also explored in this chapter. Chapter 3 describes the methodology used and research approach; experimental procedure and setup. Chapter 4 explores the test results and explains these results with analysis and discussion of whole experimental work. Chapter 5 summarizes the conclusion from this work and provides recommendations for future work.

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