



**UNIVERSITI PUTRA MALAYSIA**

***EFFECT OF ROLLING THIN FILM OVEN-AGED BINDERS AND AGING  
METHODS ON THE PERFORMANCE OF HOT MIX ASPHALT***

**RANA AMIR YOUSIF**

**FK 2017 109**



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By

**RANA AMIR YOUSIF**

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

**August 2017**

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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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By

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**August 2017**

**Chairman : Professor Ratnasamy Muniandy, PhD**  
**Faculty : Engineering**

Age hardening of bitumen has long been perceived as one of the main factors that can significantly affect the durability of bituminous paving materials. When the bitumen is age-hardened, the asphalt mixture will become brittle and its ability to support traffic-induced stresses and strains may significantly reduce. Deterioration of the pavement by readily-induced cracking may follow. In addition, excessive hardening can also weaken the adhesion between the bitumen and aggregate, resulting in loss of materials at the surface layer and generating weakening of the asphalt mixture. Furthermore, the performance of asphalt pavements is found to be very poor in moisture induced situations.

Considering this a lot of work has been done on the use of additives in bituminous mixtures as well as on modification of bitumen but none has studied the possibility of benefiting of this weak point. Research has indicated that the addition of polymers to asphalt binders helps to increase the interfacial cohesiveness of the bond between the aggregate and the binder resulting in enhanced properties of the asphalt pavements to help these increased traffic demands. However, the additive that is to be used for modification of mix or binder should satisfy both the strength requirements as well as the economic aspects. On the other hand, the properties of binders in asphalt mixtures change over time during mixing, transportation, and construction whereby as time increases, the stiffer and more brittle the binder becomes.

It is generally agreed that aging is primarily associated with bitumen oxidation and the loss of volatile/oily components from the bitumen to the air and/or aggregates during asphalt mixture production (short-term aging) and in-place service period (long-term aging). Both factors cause an increase in viscosity (or stiffness) of the bitumen and consequential stiffening of the asphalt mixture. However, in terms of the effects of the RTFO aging method on asphalt binder aging, studies have shown that it is not only

the exudation part of bitumen aging that appears to be of significance, the age time may have added effects on the aging of the asphalt binder.

This thesis attempts to provide an improved understanding of the effects of aging methods and aging time on binder and mixture aging. Ethylene-vinyl acetate (EVA) was used to modify the asphalt binder, and asphalt mixtures were artificially aged in the laboratory at five ageing times. The tensile strength and fatigue crack resistance of the mixtures, the rheological properties and chemical composition of aged asphalt binders were determined after different durations of ageing. There were compared to EVA-modified asphalt binder and mixtures at five different proportions. This asphalt binder and mixtures ageing study, utilized two different ageing simulation methods (Short-Term Oven Ageing, STOA) with five ageing times of 45, 85, 125, 165, and 205 min and a new laboratory technique. Short-Term Steam Ageing (STSA) for binder aging by using Steam Cooker. This new technique was used to study the effect of steam and heat on asphalt during storage and asphalt pavement during its service life. Five steam aging duration (0, 15, 30, 45, 60, and 75min) with four steam aging stages at different pressure and temperature for each stage were selected to investigate the physical, rheological properties of steam-aged asphalt binder. Results of the study were subjected to comparative analysis in order to determine the effect of aging time and aging method on the test results in order to have a better understanding of the mechanism of asphalt binder oxidation, which is the main cause of asphalt age hardening Hence, a suitable asphalt binder aging time could be selected for asphalt mixtures in order to show clearer effects of aging time and aging method. Through physical, rheological and mixture tests the following results have been reached:

1. These methods show a well-defined condition and best performed using 2-holes opening with a pressure of 1.10 Milibar and a temperature of 98.8 for 30-minute conditioning time.
2. Increase in viscosity and inoculation in homogenous permeability in case of 2, 3 and 4 halls as compared with 1 hall due to the pressure disturbance of water drainage from the wall openings.
3. Through comparative results of TSR test for the four cases with the limits of specifications, the investigation found that 2-holes give the best result as compared to the other situation.

Using RTFO Aged Binder instead of EVA Modified Binder is suitable for some cases especially for physical and rheological asphalt binder test.

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

**KESAN PENUAAN BAHAN PENGIKAT ROLLING THIN FILM  
OVEN- AGED(RTFO) DAN KAEDAH PENUAAN KE ATAS SIFAT-SIFAT  
CAMPURAN ASFALT PANAS**

Oleh

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Pengerasan bitumen telah lama dilihat sebagai salah satu faktor utama yang boleh menjejaskan ketahanan bahan-bahan turapan bitumen. Apabila bitumen mengeras, campuran asfalt akan menjadi rapuh dan keupayaannya untuk menampung tekanan lalu lintas, yang boleh meningkatkan tegasan dan terikan, mungkin berkurangan dengan ketara. Kemerosotan turapan akibat keretakan sedia ada akan berlaku. Di samping itu, pengerasan berlebihan juga boleh melemahkan lekatan antara bitumen dan agregat, menyebabkan kehilangan bahan-bahan pada lapisan permukaan dan seterusnya menjana kelemahan campuran asfalt tersebut. Prestasi turapan asfalt juga didapati sangat lemah dalam situasi yang dipengaruhi oleh kelembapan.

Berdasarkan faktor-faktor tersebut di atas, banyak kajian telah dilaksanakan ke atas penggunaan bahan aditif dalam campuran dan pengubahsuaian bitumen. Kajian telah menunjukkan bahawa penambahan polimer kepada pengikat asfalt membantu meningkatkan kejeleketan antara agregat dan pengikat di mana ini boleh meningkatkan sifat-sifat turapan asfalt untuk memenuhi keperluan penggunaan turapan. Walau bagaimanapun, bahan aditif yang akan digunakan dalam campuran dan pengubahsuaian bitumen bukan sahaja murah tetapi kuat. Hakikatnya, sifat-sifat pengikat dalam campuran asfalt boleh berubah mengikut masa, iaitu semasa bancuhan, pengangkutan dan pembinaan. Bahan-bahan pengikat akan mengeras dan rapuh mengikut masa.

Secara umumnya dipersetujui bahawa penuaan pada dasarnya dikaitkan dengan pengoksidaan bitumen dan kehilangan komponen meruap/berminyak daripada bitumen kepada udara dan/atau agregat semasa pengeluaran campuran asfalt (penuaan jangka pendek) dan jangka masa keberadaan di tempat perkhidmatan (penuaan jangka panjang). Kedua-dua faktor ini menyebabkan peningkatan kelikatan (atau kekakuan)

bitumen yang membawa kepada kekakuan campuran asphalt itu. Walau bagaimanapun, kajian telah menunjukkan bahawa kesan dari kaedah penuaan RTFO ke atas penuaan pengikat asphalt adalah bukan sahaja pada penuaan pelelehan bitumen, masa menua juga merupakan kesan tambahan yang tidak boleh diabaikan dalam penuaan pengikat asphalt.

Tujuan tesis ini adalah untuk memberi pemahaman yang lebih baik mengenai kesan-kesan kaedah penuaan dan masa penuaan ke atas pengikat dan campuran. Ethylene-vinyl acetate (EVA) telah digunakan untuk mengubahsuai pengikat asphalt dan campuran-campuran asphalt yang dituakan secara buatan dalam makmal melalui lima jangkamasa penuaan. Kekuatan tegangan dan rintangan retak lesu campuran, sifat-sifat reologi dan komposisi kimia pengikat asphalt yang dituakan ditentukan selepas jangka masa penuaan yang berbeza dan dibandingkan dengan pengikat asphalt yang telah diubahsuai dengan EVA dan campuran-campuran dengan lima nisbah yang berbeza. Kajian penuaan pengikat asphalt dan campuran dijalankan melalui dua kaedah simulasi penuaan yang berbeza iaitu penuaan ketuhar jangka pendek (STOA) dengan lima jangkamasa penuaan sepanjang 45, 85, 125, 165, dan 205 minit dan teknik terbaru Penuaan Jangka Pendek Stim (STSA) dengan lima jangka masa penuaan 15, 30, 45, 60, dan 75 minit. Teknik baru ini adalah untuk mengkaji kesan stim dan kepanasan ke atas asphalt yang disimpan dan atas permukaan jalan asphalt sepanjang tempoh penggunaannya. Lima tempoh penuaan stim (0, 15, 30, 45, 60, and 75min) dengan empat peringkat penuaan stim pada tekanan dan suhu yang berbeza pada setiap peringkat dipilih untuk mengkaji ciri-ciri fizikal, dan reologi pengikat asphalt melalui kaedah penuaan stim. Keputusan kajian ini tertakluk kepada analisis perbandingan untuk menentukan kesan jangka masa penuaan dan kaedah penuaan agar mekanisme pengoksidaan bitumen, yang merupakan punca utama pengerasan penuaan asphalt, boleh difahami dengan lebih baik dan jangka masa penuaan pengikat asphalt yang sesuai boleh dipilih. Melalui ujian fizikal, reologikal dan campuran, keputusan yang diperolehi adalah seperti berikut:

1. Bahawa keadaan dan kaedah terbaik adalah melalui bukaan 2 lubang dengan tekanan 1.10 Milibar dan suhu 98.8 selama 30 minit tempoh penyesuaian.
2. Peningkatan kelikatan dan inokulasi dalam ketelapan homogen dalam kes 2, 3 dan 4 ruangan berbanding 1 ruangan, disebabkan oleh gangguan tekanan akibat aliran air dari bukaan dinding.
3. Dalam perbandingan keputusan ujian TSR untuk keempat-empat kes dengan had-had spesifikasi tertentu, kajian menunjukkan 2 ruangan memberikan keputusan yang terbaik berbanding situasi-situasi lainnya.

## ACKNOWLEDGEMENTS

In the Name of Allah, Most Gracious, Most Merciful, all praise and thanks are due to Allah, and peace and blessings are upon His Messenger. First of all, I would like to express my sincere thanks and appreciation to my supervisor Professor Dr. Ratnasamy Muniandy for his support, thoughtful guidance, encouragement, and help throughout the course of my study. I would like to thank Professor Madya Ir. Salihudin Hassim and Dr. Fauzan Mohd Jakarni for being on my supervisor committee. Their cooperation, guidance, and patience have been invaluable.

I am indebted to many people for their kindness and effort on my behalf. Foremost among them is Dr. Ratnasamy, my supervisor, whom I wish to thank for his kind advice and patient mentorship. The constancy of his support has allowed me to fulfill my objectives in graduate school, and without him, this program would not have been accomplished.

I want to express my gratitude to Iraqi Government for their support during my study period and for giving me this opportunity. Also, I would like to express my gratitude to my university (Al-Mustansiriyah University) management and staff for their help.

Finally, all my gratitude goes to my family above all my mother Mona Salih, husband Ahmed Mohammed Ali, and children Mayar, Yousif, Mohamed for their love and support which have always encouraged me to pursue. This work was possible because of their dedication and a strong commitment to my education and because of their high expectations and belief in me to achieve my goal.

Last but not least, my very special thanks are also given to all my research friends who were, directly and indirectly, involved in this research and with whom I have shared many enjoyable times.



I certify that a Thesis Examination Committee has met on 14 August 2017 to conduct the final examination of Rana Amir Yousif on her thesis entitled "Effect of Rolling Thin Film Oven-Aged Binders and Aging Methods on the Performance of Hot Mix Asphalt" 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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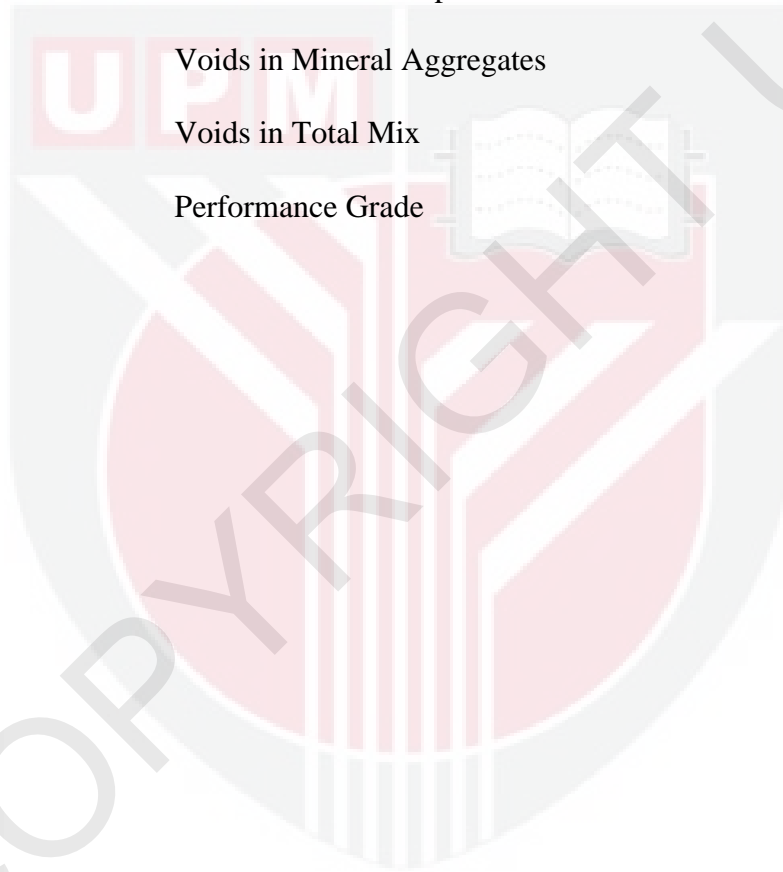
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## LIST OF ABBREVIATIONS

AASHTO	American Association of State Highway and Transportation Officials
AIV	Aggregate Impact Value
ASCE	American Society of Civil Engineering
ASTM	American Society for Testing and Materials
BS	British Standard
DSR	Dynamic Shear Rheometer
EVA	Ethylene -vinyl acetate
FHWA	Federal Highway Administration
GTM	Gyratory Testing Machine
HMA	Hot Mix Asphalt
IDT	Indirect Tensile Test
ITFT	Indirect Tensile Fatigue Test
LA	Los Angeles Abrasion
LVDT	Linear Variable Differential Transducer
LWT	Loaded Wheel Tracking
MATTA	Material Testing Apparatus
NAPA	National Asphalt Pavement Association
NCAT	National Center for Asphalt Technology
NCHRP	National Cooperative Highway Research Program
OAC	Optimum Asphalt Content
PAV	Pressure Aging Vessel
RPM	Revolutions per minute
RTFO	Rolling Thin Film Oven

SGC	Superpave Gyrotory Compactor
SHRP	Strategic Highway Research Program
SSD	Saturated Surface Dry
Superpave	Superior Performing Asphalt Pavement
TMD	Theoretical Maximum Density
UPM	Universiti Putra Malaysia
VFA	Voids filled with Asphalt
VMA	Voids in Mineral Aggregates
VTM	Voids in Total Mix
PG	Performance Grade





# CHAPTER 1

## INTRODUCTION

### 1.1 Background

Asphalt pavements suffer from fatigue cracking and thermal cracking, aggravated by oxidation and hardening of asphalt. This negative impact of asphalt oxidation on pavement performance has not been considered adequately in pavement design. No doubt, pioneering pavement engineers soon realized that in the short-term asphalt hardened after heating, mainly due to volatilization, and, in the long term it hardened, mainly due to oxidation.

Hardening is primarily associated with loss of volatile components in asphalt during the construction phase (short-term aging), and progressive oxidation of the in-place material in the field (long-term aging). Both factors cause an increase in viscosity of the asphalt and a consequent stiffening of the mixture. This may cause the mixture to become hard and brittle and susceptible to disintegration and cracking failures. Also, the products of oxidation may render the mixture less durable than the original mixture, in terms of wear resistance and moisture susceptibility. However, "aging" is not necessarily negative phenomenon, since some aging may help a mixture achieve optimum properties.

With regard to asphalt mixtures, aging is associated with the phenomenon of hardening. Two other terms commonly used are "age hardening" and "embrittlement". The two major aging processes that affect the asphalt and consequently its properties and performance are; short term and long term aging. Short-term aging occurs during the construction phase and is primarily due to oxidation and to the loss of volatile components while a mix is hot and this is a relatively rapid process. Long-term aging is primarily due to the progressive oxidation of a mixture while it is in service. Other factors that can contribute to aging of in-place asphalt materials include steric hardening (molecular re-structuring) and actinic light (mainly ultraviolet radiation). These processes are rather slow and tend to take place over a relatively long time. Additionally, they can be affected by the mix volumetric properties such as air voids content (e.g., higher air voids content results in higher oxidation and more hardening of the mix), and even the location of the asphalt layer in the pavement system (e.g., asphalt layers placed deeper in the pavement section age considerably much slower than a surface layer directly exposed to air). Because it is the binder that ages in the mix, most studies on aging have focused on studying the aging performance characteristics of the binder. However, some studies have also investigated the aging properties of asphalt mixtures which provide a more realistic investigation that considers the influence of all variables on aging. Assess the effects of aging on both asphalt and mixture became necessary and urgent because it directs impact on both characteristics of asphalt and the acted of the asphalt mixture. EVA modifier was selected to compare it with aged binder because this type of additive has a positive

effect on viscosity as well as aged binder also possible to know the properties of this type of additive.

## 1.2 Problem Statement

Bitumen's are widely used in road construction largely because they are relatively inexpensive and generally provide good durability in paving mixtures. In addition, bituminous pavements are generally characterized by their immediate serviceability, good riding quality, and absence of joints. As an organic substance, bitumen can age harden, i.e. increase in viscosity, which results in significant deterioration of the serviceability of bituminous paving materials. When the bitumen is age hardened, the asphalt mixture will become brittle and its ability to support traffic-induced stresses and strains may significantly reduce, leading to deterioration of the pavement by readily-induced cracking. In addition, excessive hardening can also weaken the adhesion between the bitumen and aggregate, resulting in loss of materials at the surface layer and generate weakening of the asphalt mixture. Much effort has been afforded to the study of age hardening and many factors have been established to be capable of affecting bitumen aging.

Many of the plants that produce asphalt tried to exploit the increase occurring in viscosity to sale asphalt that lose some of the service life and inflatable as pure asphalt. This action led to the appearance of aging sign on newly constructed roads through the appearance of cracks, wearing of service and stripping. For this reasons the need to study and re-evaluation of aged asphalt become an urgent need. Otherwise, utilization this positive characteristic (increase in viscosity) value in marking these materials that loss of some of it is service age as a good article and within the approved international standard.

Because of aging, the asphalt mixture will become brittle and its ability to support traffic-induced stresses and strains may significantly reduce. Deterioration of the pavement by readily-induced cracking may follow. In addition, excessive hardening can also weaken the adhesion between the bitumen and aggregate, resulting in loss of materials at the surface layer and generate weakening of the asphalt mixture. For these reasons, two methods were selected to aged asphalt binder within the service life of it. Two different aging times depend on aging/time and aging/moisture was select to compare the effect of aging on asphalt binder properties in both rutting and fatigue distress.

This thesis attempts to provide an improved understanding and was initiated to compare the effects of different aging methods and aging durations of on asphalt binder and mixture aging also presents a new laboratory technique for binder aging by using Steam Cooker. A new technique was used to study the effect of steam and heat on asphalt during storage and asphalt pavement during its service life. Five steam aging duration (0, 15, 30, 45, 60, and 75min) with four steam aging stages at diffrent pressure and temperature for each stage were selected to investigate the physical, rheological properties of steam aged asphalt binder. A pure asphalt binder and

Ethylene-Vinyl Acetate (EVA) modified binder and mixture aging study, utilising two different aging simulation methods, Asphalt binder and mixtures unmodified and EVA modified were artificially aged at five different durations in the laboratory and the stiffness, tensile strength, fatigue performance of the mixtures, the rheological properties and chemical composition of recovered binders were determined after different durations of aging and a study on the effect of aging durations on asphalt binder and mixture aging were carried out to provide and to demonstrate the beneficial impact of aging time and EVA modifications on aging characteristics of asphalt binder and mixture. This is so that the mechanism of binder and mixture oxidation, which is the main cause of asphalt binder age hardening, could be better understood and a suitable binder aging time could be selected for both asphalt binder and mixtures to show the effects of time on aging more clearly.

### **1.3 Research Objectives**

1. To evaluate the aging effects of Short-Term Oven Aging (STOA) and Long-Term Oven Aging on laboratory prepared blown asphalt, EVA modified binder and Steam aging asphalt binders.
2. To determine the tensile strength and fatigue performance of blown and EVA modified asphalt binder on HMA mixtures.
3. To determine the sensitivity of Steam Aged asphalt binder and mixture.

### **1.4 Hypothesized effects of aging methods and duration**

Based on literature review and prior experience, the working hypothesis for this study was that, within practical ranges, using EVA with large quantity and or high aging duration causes an increase in viscosity of the asphalt and a consequent stiffening of the mixture. This may cause the asphalt binder and mixture to become hard and brittle and susceptible to disintegration and cracking failures which affect pavement performance when premature distress is a concern. Also, the products of oxidation may render the mixture less durable than the original mixture, in terms of wear resistance and moisture susceptibility. The following discussion suggests a plausible rationale.

In increasing asphalt content means decreased asphalt binder/or mixture viscosity, the asphalt binder /or asphalt concrete mixture becomes relatively soft and will be more viscous especially at high temperature and thus unable to resist the imposed load and incapable of resisting the resulting shear strain., However, if the modifier proportion/and or aging time is extremely low, it may extend the asphalt cement, making it act like a higher asphalt content, and lower the Marshall Stability Index and Resilient Modulus. Moreover, asphalt mixtures with high asphalt content are associated with high cost. The higher proportion/and higher aging time absorb the oily part of asphalt structure (maltenes) which in return increases the viscosity of the modified asphalt binder /asphalt mixture and it makes the asphalt binder /asphalt mixture more stiffer resulting in considerably lower strain when compared with the unaged/and or unmodified asphalt binder /asphalt mixture and this enhancing the temperature susceptibility and increasing the Marshall Stability Index and Resilient

Modulus values and in return enhance the asphalt binder /asphalt mixture performance at high temperature and prevent the occurrence of premature pavement distresses.

### **1.5 Significance of the Study**

The new knowledge from this research can be used to develop asphalt mixes with superior resistance to modes of pavement degradation. In addition, results that will contribute to a better understanding of the effects of EVA modifier and aging time on the performance properties of asphalt binder and HMA mixtures which could save the agencies and organization a good sum of money on maintenance and rehabilitation work on our roads.

The contribution of this research, therefore, is to clarify the influence of aging method and aging time on asphalt binder and asphalt pavements particularly HMA mixtures and to demonstrate advanced test methods that can be used in the performance evaluation of various asphalt binder and mixtures. With additional validation and calibration, the comprehensive methodology described herein and the research outcomes may serve as the foundation bases for improving a performance-based HMA Mix design and performance-related HMA specifications. Also, the knowledge gained from this research would be transferable to the use of other unconventional materials in asphalt and benefit the highway construction and rehabilitation industry as well as the overall society.

### **1.6 Thesis Layout**

The research work is organized into five chapters. Chapter I has provided a brief background to research on aging of bitumen, describe the statement of the problem, and the hypothesis of the study with special emphasis on the objectives of the proposed study.

**Chapter 2** reviews and discusses the background information, the available literature relating to the constituent materials; bitumen, Ethylene-Vinyl Acetate (EVA) and its application in asphalt mixtures, a review of bituminous mixtures including testing methods to study mechanical properties are discussed. Furthermore, description of short-term and long-term aging was reviewed in this chapter. Also, details for beam fatigue and indirect tensile test (moisture susceptibility) were explored.

**Chapter 3** provides information about the materials used, sample preparation, the experimental procedures, research approach, and the experimental organization developed.

**In Chapter 4** presents full details of test results and analytical discussions. Statistical analyses including regression models are also included in this chapter.

Finally, a summary of the findings of this study, conclusions and some recommendations for future research are presented in Chapter 5. Appendixes are attached at the end of this thesis, which contains all raw data used in this research.



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