

การทดสอบระบบไอทีซิสการทดสอบระบบไอทีซิส
Test of iThesis System Test of *iThesis* System

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Noppawan Kunanusorn

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หัวข้อ	การทดสอบระบบไอทีลิสการทดสอบระบบไอทีลิส
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การขนส่งยางมะตอยในปัจจุบันนิยมส่งแบบของเหลวซึ่งจำเป็นต้องให้ความร้อนตลอดระยะเวลาที่ขนส่งซึ่งสิ้นเปลืองพลังงาน และจำเป็นต้องใช้รถบรรทุกพิเศษในการขนส่ง การขนส่งในลักษณะกึ่งแข็งที่มีบรรจุภัณฑ์ห่อหุ้มนอกจากจะมีข้อดีกว่าในเรื่องการประหยัดพลังงาน ยังสามารถใช้รถบรรทุกธรรมดาขนส่งได้ จุดประสงค์ของงานวิจัยนี้ คือ การพัฒนาบรรจุภัณฑ์ที่ทนร้อนขณะบรรจุและสามารถหลอมผสมไปกับยางมะตอยได้โดยไม่ก่อให้เกิดขยะและไม่ทำให้สมบัติของยางมะตอยเสียไป โดยงานวิจัยนี้แบ่งเป็น 2 ส่วน คือ (1) การพัฒนาบรรจุภัณฑ์จากเทอร์โมพลาสติกวัลคาไนเซต (thermoplastic vulcanizate, TPV) ของยางธรรมชาติ (natural rubber, NR) ร่วมกับเอทิลีนไวนิลอะซิเตดโคพอลิเมอร์ (ethylene vinyl acetate copolymer, EVA) จากนั้น (2) จะนำ TPV ที่ได้ไปทดสอบบรรจุยางมะตอยและนำไปหลอมผสมกับยางมะตอย พร้อมกับทดสอบสมบัติของยางมะตอย และศึกษาการปรับปรุงสมบัติยางมะตอยด้วย TPV ที่มีระดับการเชื่อมขวาง (degree of crosslink) ที่แตกต่างกัน โดยในส่วนที่ 1 เป็นการศึกษาวิธีการผสม TPV ที่แบ่งเป็น 2 ขั้นตอน คือ การเชื่อมขวางแบบพลวัต (dynamic vulcanization, DV) และการผสมภายหลัง (further blending, FB) กำหนดอัตราส่วนโดยน้ำหนักของ EVA:NR เท่ากับ 50:50 เลือกใช้สารเชื่อมขวางเป็นไดคิลมิลเพอร์ออกไซด์ (dicumyl peroxide, DCP) ที่ปริมาณ 2 ส่วนใน 100 ส่วนของเรซิน (part per hundred resin, phr) และสารป้องกันการเสื่อมสภาพ (thermal stabilizer, TS) ปริมาณ 1 phr โดยศึกษาวิธีผสม 3 วิธี ได้แก่ NR-DV คือ DV เฉพาะ NR และ FB EVA กับ TS, Split-DV คือ DV EVA บางส่วนกับ NR และ FB EVA ส่วนที่เหลือ กับ TS, และ All-DV คือ DV EVA ทั้งหมดกับ NR และ FB เฉพาะ พบว่าวิธีการผสมส่งผลต่อสมบัติของ TPV โดยวิธี NR-DV ให้ชิ้นงานที่ไม่เป็นเนื้อเดียวกันเนื่องจากการมี NR เชื่อมขวางกันเองมากเกินไป ไม่สามารถนำไปใช้งานต่อได้ ส่วน All-DV มีสมบัติเชิงกลดีกว่า Split-DV เนื่องมาจากการเกิดการเชื่อมขวางกันเองของ EVA มากเกินไป จึงนำวิธีผสม Split-DV ไปเตรียมชิ้นงานสำหรับการทดสอบบรรจุภัณฑ์ยางมะตอย โดยปรับระดับการเชื่อมขวางของ TPV โดยการปรับปริมาณ DCP ตั้งแต่ 0 – 1.5 phr โดยผสม TPV กับยางมะตอยที่อัตราส่วนร้อยละ 5 โดยน้ำหนักเพื่อทำยางมะตอยที่ดัดแปรด้วยพอลิเมอร์ (polymer modified asphalt, PMA) พบว่าปริมาณการเชื่อมขวางส่งผลต่อสมบัติของ PMA โดยเมื่อมีการเชื่อมขวางมาก

จะทำให้ยางมะตอยที่ได้มีความทนทานมากขึ้น แต่จะมีความหนืดเมื่อหลอมเหลวน้อยกว่าการเติม TPV ที่ไม่เชื่อมขวาง เนื่องมาจากการต้านการไหลของสายโซ่พอลิเมอร์ที่เกิดการเกี่ยวพันกัน (entanglement) มีผลในการต้านการไหลมากกว่าอนุภาคยางเชื่อมขวางที่แขวนลอยในยางมะตอย



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Asphalt is normally delivered in liquid form which was heated all the time. It consumes more energy and need the special truck to deliver it. An alternative way to deliver the asphalt in form of solid covered with package can reduce energy consumption and normal truck can be used to deliver. The objective of this research was to develop the zero-waste package for asphalt that was no waste after use and not harmful to asphalt properties. The research consists of two parts which are (1) development of package from natural rubber (NR) and ethylene vinyl acetate copolymer (EVA) thermoplastic vulcanizates (TPV), and (2) Test of asphalt package prepared from the TPV and studied on asphalt modification by incorporating TPV with various degree of crosslink. For the first part, the mixing method consists of two steps: dynamic vulcanization (DV) and further blending (FB). The weight ratio of EVA/NR was fixed at 50/50 wt/wt. Dicumyl peroxide (DCP) was used as a crosslinking agent and was fixed at 2 phr. Thermal stabilizers (TS) were fixed at 1 phr. Three mixing methods were compared: NR-DV which NR was only DV and then FB with EVA and thermal stabilizers (TS), Split-DV which NR and some EVA were DV and then FB with the rest of EVA and TS, and All-DV which NR and EVA was DV and then FB with TS. It was found that mixing method affected properties of TPV. NR-DV sample showed a heterogeneous texture because there was only crosslinked NR, this sample cannot be used. Mechanical properties of All-DV sample were inferior to those of Split-DV sample because of self-crosslinked EVA. The Split-DV method was selected to prepare the polymer modified asphalt (PMA). The TPV with different degree of crosslink were prepared by varying the DCP content from 0 to 1.5 phr. The TPV content was fixed at 5 wt%. It was found that the degree of crosslink affected the properties of PMA. The more degree of crosslink of TPV improved the physical

properties of PMA with less viscosity when compared with the asphalt modified with non-crosslink TPV. It might be due to the long-chain polymer contained in non-crosslink TPV which has more entanglement of chain than the short-chain polymer or crosslinked rubber particles.



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นพวรรณ คุณานุสรณ์



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CHAPTER I

INTRODUCTION

1.1 General Introduction

Asphalt is a mixture of hydrocarbons and is commercially obtained from petroleum refinery process. It is solid at room temperature while it becomes liquid and can be pumped when its temperature is higher than 120 °C (Manjunath H. N., Ramesh Babu N., Suhas Kumar S., Sushanth H. Gowda, & Kiran Aithal S., 2017; Speight, 2016). The asphalt is widely used as a binder in paving (road construction), roofing (asphalt shingles) and sealing application. Normally, the asphalt is mixed with aggregates at temperature around 160 – 180 °C. This asphalt is called a hot-mix asphalt. But, due to the environmental issue, there is demand for lower organic compound emission during mixing of asphalt and aggregates. This can be done by lowering the mixing temperature of asphalt and aggregates to be around 130 °C (Ahmed, Hesp, Paul Samy, Rubab, & Warburton, 2012; Rubio, Martínez, Baena, & Moreno, 2012; Yu, Leng, Zhang, Li, & Zhang, 2020). Therefore, a warm-mix asphalt is interesting.

In general, the temperature of asphalt obtained from the refinery process is about 200 °C. After it is transferred to storage tank, the temperature of asphalt drops to around 160 °C. It needs to be maintained at the temperature not lower than 160 °C so that it can be pumped into the special tank truck. To counter the heat loss, during the delivery process, the tank of asphalt is continuously heated to maintain the asphalt in liquid state. After delivery process, the asphalt is either mixed with the aggregates at a hot-mix plant or kept in the storage tank with heating unit. However, the asphalt cannot be kept more than three days at high temperature because of thermal aging (Ahmed et al., 2012; Xu et al., 2017). Moreover, the special truck as shown in Figure 1.1 is required to deliver the bulk liquid asphalt to a hot-mix plant shown in Figure 1.2.



Figure 1.1 Special truck for asphalt delivery process (Tipco Asphalt Public Company Limited)



Figure 1.2 Hot mix plant (Falagroup)

To reduce the energy consumption during delivery and storage of asphalt, the asphalt should be delivered and kept in a solid form that can be delivered by using a normal truck at ambient temperature. However, due to the tackiness of solid asphalt, it should be covered with some plastic film which can be melted with asphalt without causing harm to the asphalt. This type of plastic film or package is called a zero-waste package. The proper asphalt package should consist of three functions: (i) endure heat during hot asphalt filling, (ii) good mechanical properties during delivery process, and (iii) able to melt with asphalt during mixing with hot asphalt without causing harm to the asphalt. The asphalt package that can be melted with asphalt was first invented in 1999. It has since been improved using many techniques to overcome its limitations (Chehovits & Glover, 2013; Marchal, 2007; Vermilion et al., 1999). More works are still needed.

Nowadays, a major environmental concern is plastic waste, especially single-use plastic packaging. Many countries provide policies to solve this problem such as the reduce-reuse-recycle or 3R waste management (Conserve Energy Future). Thailand is ranked 6th in the world for mismanagement of plastic waste (equaled to 1.03 million tons per year being released to the ocean) (Jambeck et al., 2015). According to the Thailand's roadmap on plastic waste management (Pollution Control Department, 2020), plastic bags with thickness lower than 36 micrometers and single-use plastics (i.e., straws, tablewares, food boxes) must be reduced and banned within 2022. Moreover, the plastic waste must be 100% recycled within 2027. To support this roadmap, the plastic waste is recommended for producing the zero-waste asphalt package. Most of plastic packaging are produced from polyethylene (PE) and its copolymer which is an ethylene vinyl acetate copolymer (EVA). The advantages of EVA are high impact and puncture resistances and it is more transparent than PE. However, the melting temperature of EVA is around 86 °C while the temperature of liquid asphalt is more than 100 °C. From this reason, the thermal resistance of EVA should be improved to endure the hot asphalt during filling.

There are many methods to improve the thermal resistance of EVA such as blending with high melting temperature or high molecular weight polymer, incorporating with rigid particles to prepare polymer composite, and crosslinking the EVA to increase the

molecular weight of polymer. In this work, blending with high melting temperature polymer as well as crosslinking were employed to increase the thermal resistance of EVA. (Davis, 2001; Fardanesh, 2006; Gregoire, 2003)

Natural rubber (NR) is an elastomeric polymer with high molecular weight ($> 100,000$ g/mol) (Kovuttikulrangsie & Sakdapipanich, 2005). Thailand is the world's biggest natural rubber production with the market share of 37% of global production (Arunmas, 2018). The price of NR has fallen since 2014 due to the excess supply. To overcome this problem, the Association of Natural Rubber Producing Countries (ANRPC) have been continuously promoting more usage of natural rubber. In addition, the NR has been used as an additive for asphalt. The road constructed with asphalt and natural rubber has longer service life than the road constructed by normal asphalt (The Rubber Economist, 2020). Due to its high molecular weight, the NR has no melting point. Thus, it cannot flow like thermoplastic at high temperature. Therefore, it has a potential to be used to increase the thermal resistance of EVA. Moreover, dynamic vulcanization technique can be applied to EVA/NR blends to transform them into thermoplastic vulcanizate (TPV) (Naskar & Babu, 2014; Samthong et al., 2019; H. Wu et al., 2016; Q. Wu et al., 2019). TPV has better thermal resistance while can be reprocessed like thermoplastic.

This dissertation consisted of two parts which were the preparation of thermoplastic vulcanizates and the fabrication of asphalt package. For the first part, the effect of mixing method and peroxide content on the properties of EVA/NR TPVs were studied. In the second part, the asphalt package from TPV with different peroxide contents were prepared and tested by filling with hot asphalt. Moreover, the properties of asphalt blending with the TPV with different peroxide content were also investigated.

1.2 Objective and Scopes

The objective of this dissertation was to develop the zero-waste package for asphalt from natural rubber (NR) and ethylene vinyl acetate copolymer (EVA) thermoplastic vulcanizates (TPV).

Thermoplastic vulcanizates (TPV) was prepared by blending EVA with NR under the proper dynamic vulcanization (DV) technique. Afterwards, the TPV was prepared as a film package and tested with hot asphalt. To evaluate the performance of film package, the test on polymer modified asphalt was performed in this dissertation. The dissertation was separated into two parts which have two sub-objectives as followed:

To study effect of mixing method and peroxide on properties of ethylene vinyl acetate copolymer and natural rubber thermoplastic vulcanizates:

EVA type 18 wt% of vinyl acetate was used.

NR of air-dried sheet type was used.

The EVA and NR content at 50 wt% each were fixed.

Dicumyl peroxide (DCP) was used as a crosslinking agent

Three mixing methods which were designated as NR-DV, Split-DV, and All-DV (the details of these three methods were described in section 4.1.2 and illustrated in Figure 4.1) were compared.

The best method providing good processability and the best mechanical properties of TPV was used to study the effect of peroxide content. The DCP content was varied from 0 to 3 phr.

To evaluate the effect of gel content of TPV on properties of EVA/NR TPV modified asphalt:

EVA and NR content at 50 wt% each were fixed.

The Split-DV method was selected to prepare the TPV

DCP content of 0, 0.5, 1, and 1.5 phr were compared.

Asphalt graded AC 60/70 was used.

TPV content of 5 wt% was fixed.

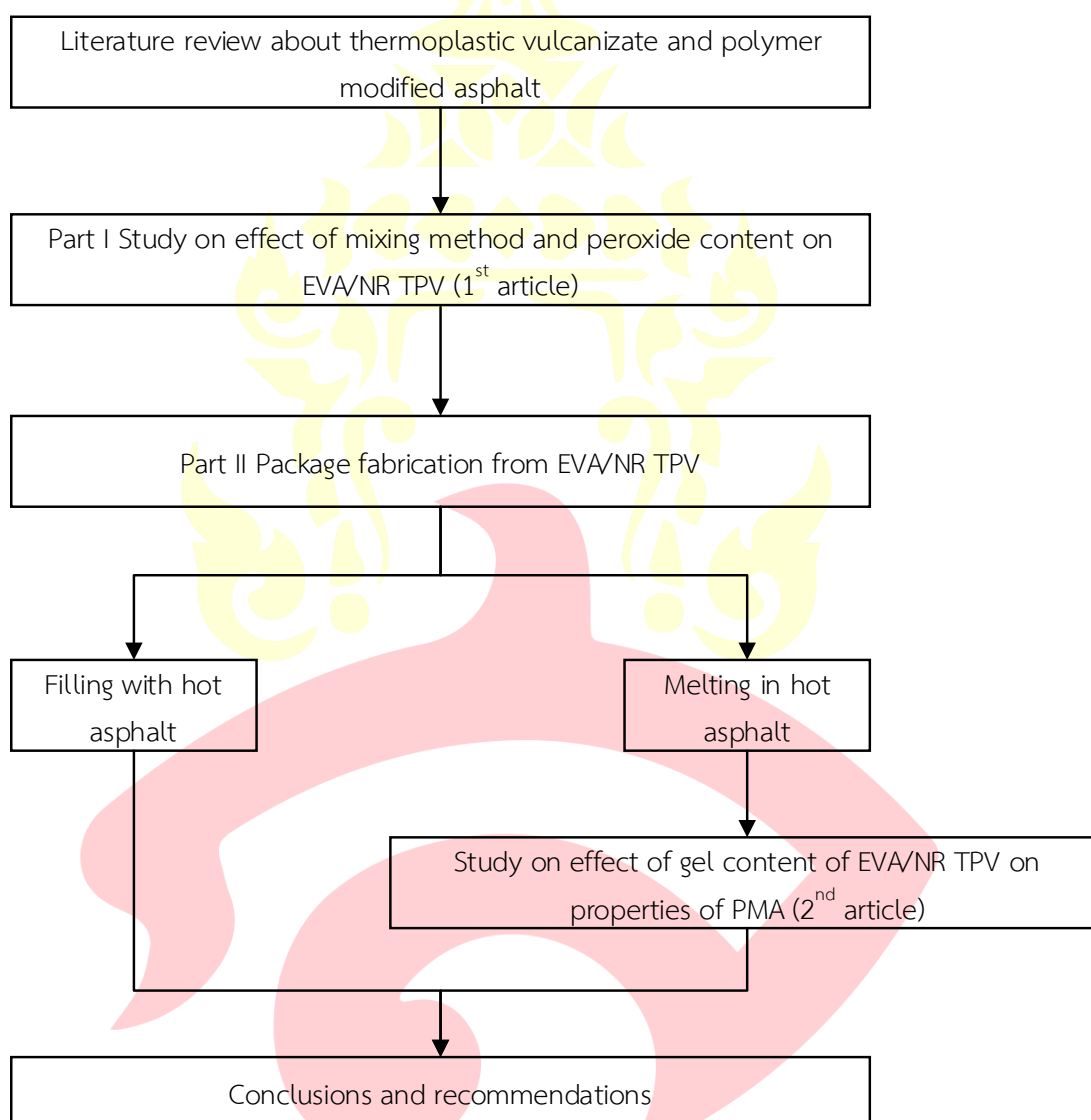
1.3 Organization of the Dissertation

The dissertation was divided into six chapters. Chapter I introduced and gave some motivation about the development of zero-waste package for asphalt and stated the objective of this research. Chapter II was a theory and literature reviews related to asphalt, patent of asphalt package, thermoplastic vulcanizate, and polymer modified

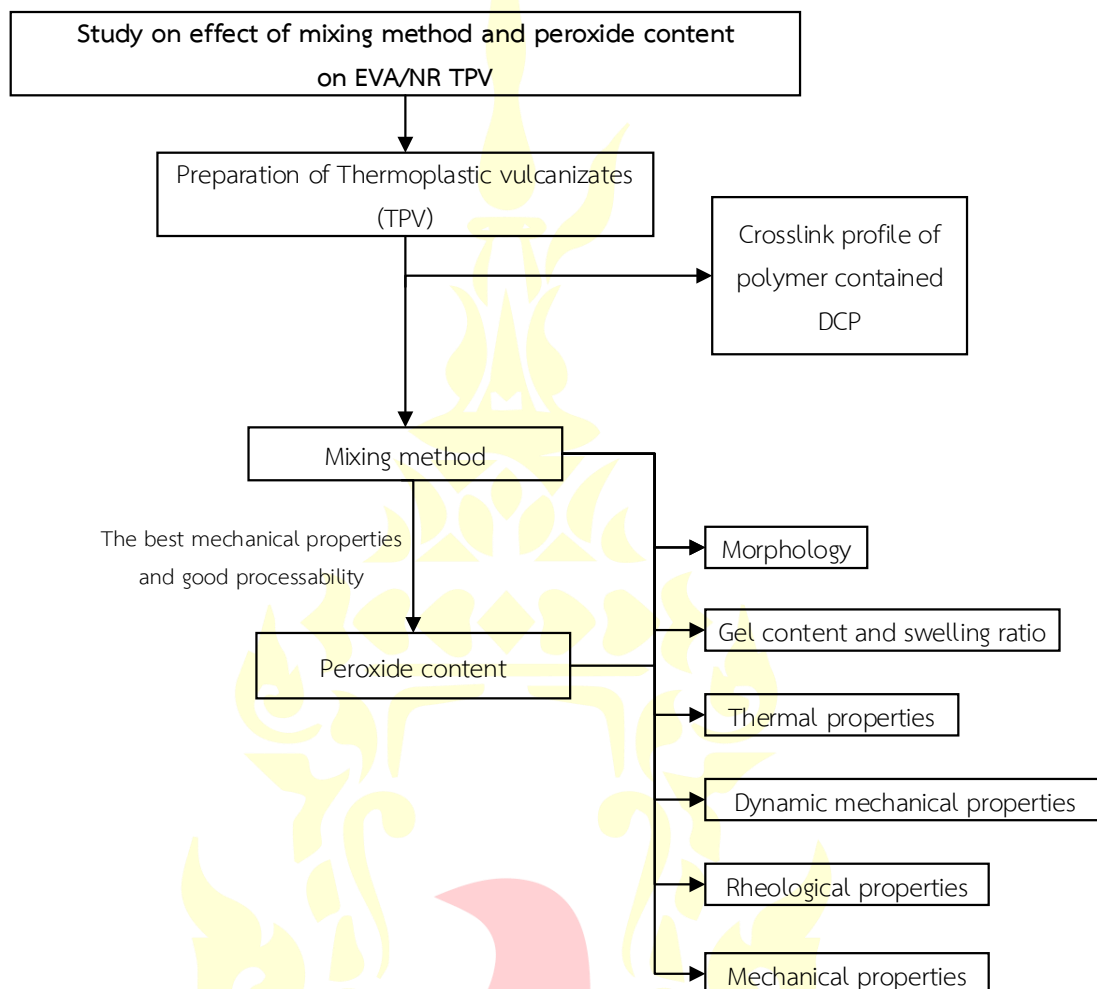
asphalt. Chapter III described the characterization techniques related to this research. Materials, sample preparation as well as results and discussion of ethylene vinyl acetate copolymer and natural rubber thermoplastic vulcanizates (TPV) were presented in Chapter IV. Consequently, the preparation and test of the package from TPV were shown in Chapter V. Finally, the Chapter VI gave the overall conclusions and recommendations for future works.

1.4 Research Methodology

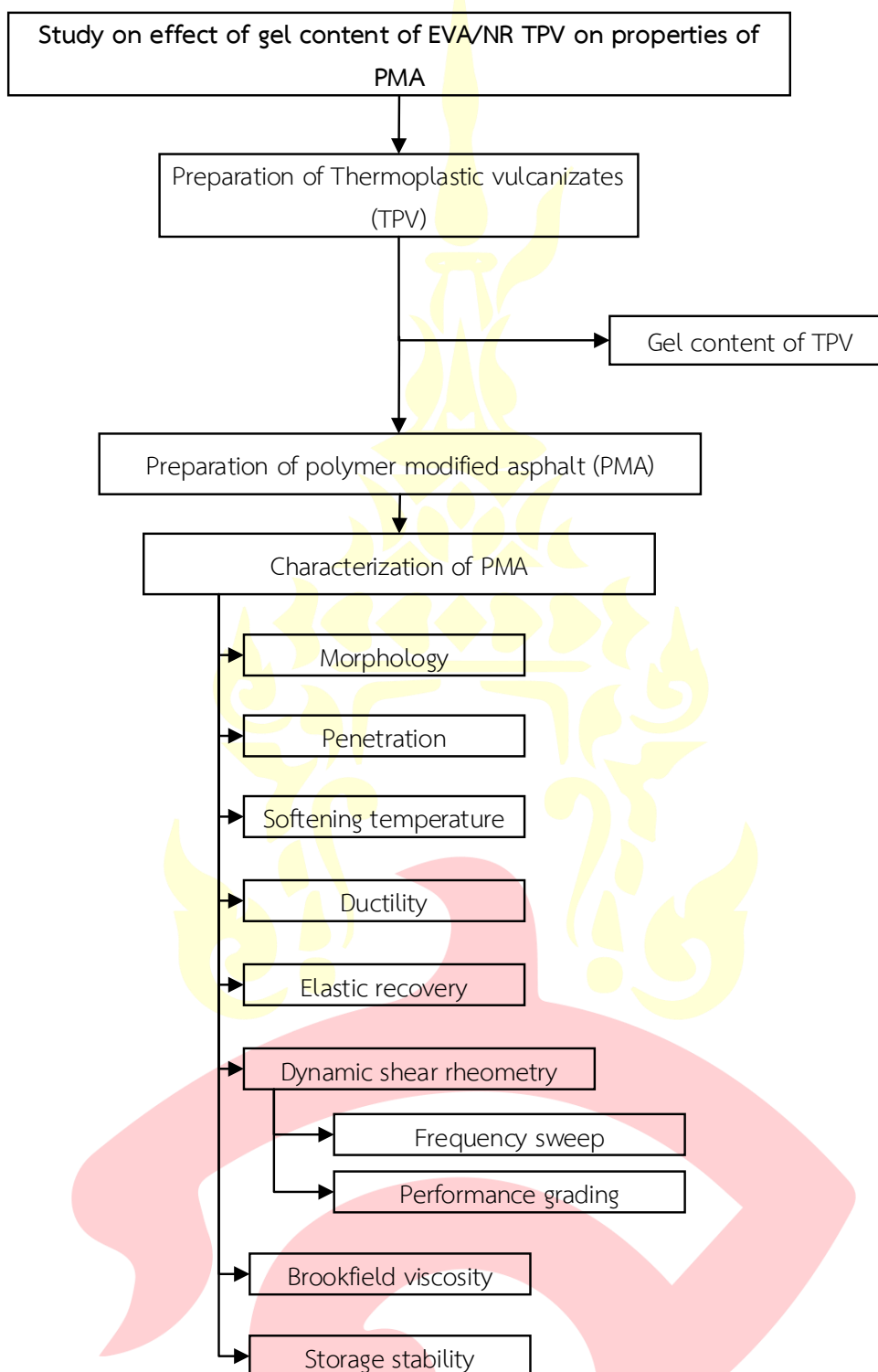
1.4.1 Overall Methodology



1.4.2 Study on Effect of Mixing Method and Peroxide Content on EVA/NR TPV



1.4.3 Study on Effect of Gel Content of EVA/NR TPV on Properties of PMA



CHAPTER II

THEORY AND LITERATURE REVIEWS

This chapter described about asphalt, patent of asphalt package, ethylene vinyl acetate copolymer, natural rubber blends, thermoplastic vulcanizates, peroxide vulcanization, and polymer modified asphalt.

2.1 Asphalt

2.1.1 General Information

Asphalt is a mixture of various hydrocarbons which the boiling temperature is more than 200 °C. It is commercially obtained from residue of petroleum refinery process. It consists of many aromatic hydrocarbons which make the asphalt color become black and it is a good binder. However, the asphalt material does not have exact molecular structure and amount of each hydrocarbon substance due to the variety of crude oil. Asphalt has been used in pavement, sealant, and roofing application due to its low-cost, thermoplastic behavior, and climate resistance (Porto et al., 2019).

Based on the processing temperature, the asphalt can be classified into three types which are hot mix asphalt (HMA), warm-mix asphalt (WMA), and cold-mix asphalt (CMA) (Rubio et al., 2012). The hot mix asphalt or HMA is a traditional asphalt which the processing temperature is higher than 160 °C while the processing temperature of warm mix asphalt or WMA is about 100 - 140 °C. The WMA was first developed in 1999 (European Asphalt Pavement Association, 2010). The viscosity of asphalt is reduced by blending with water or organic additive at temperature higher than their boiling temperatures. After the water or organic additive has evaporated, the micro voids inside the asphalt are created and the viscosity of asphalt decrease. Therefore, the processing temperature of WMA is lower than that of HMA. The benefits of low processing temperature of asphalt are low fumed emissions, reduced energy consumption, improved workability, compaction efficiency, and longer pavement life (Ahmed et al., 2012; European Asphalt Pavement Association, 2010; Vaitkus, Cygas, Laurinavicius, & Perveneckas, 2009). The CMA is an asphalt that is dispersed in media (i.e., water or organic solvent) which can be mixed with aggregate at ambient

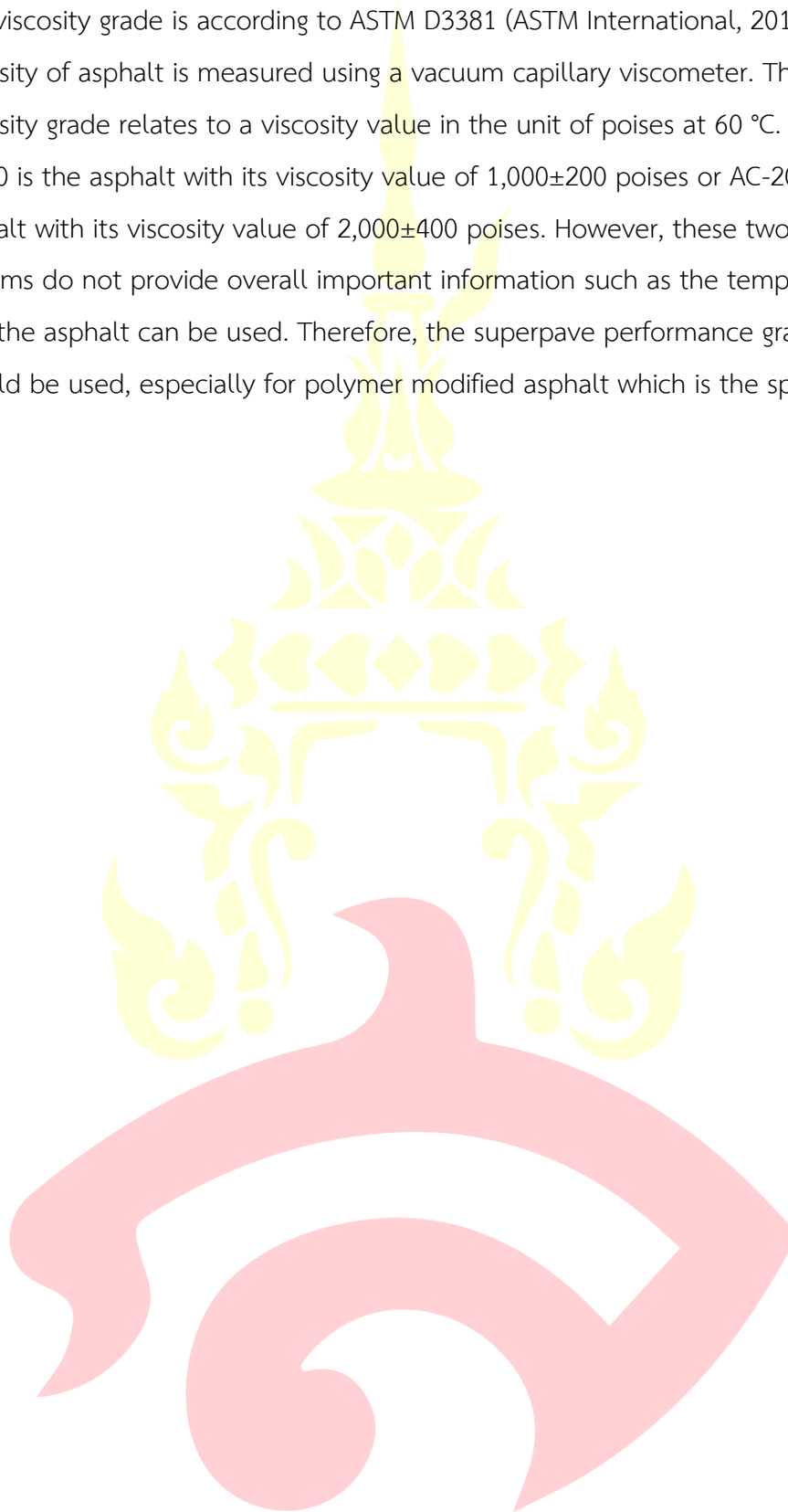
temperature. There are two types of CMA based on the media: asphalt emulsion which the media is water, and cutback asphalt which the media is organic solvent (Aimix group, 2019; Chehelgo, C. Abiero Gariy, & Muse Shitote, 2018; Kurt & Austria, 2015). The CMA is normally used for road maintenance due to easy preparation. However, the evaporation of solvent in CMA should be concerned and it can affect the properties of asphalt mixture. Due to this reason, HMA has been used until now.

2.1.2 Grading Systems

At present, there are three popular systems for determining characteristics of asphalt which are penetration grade, viscosity grade, and performance grade. The grade is specified according to the usage condition.

Penetration grade is the oldest grade and still used at present because of the simple test method. Penetration of asphalt is measured according to ASTM D946 (ASTM International, 2020). For example, the AC 40/50 means the asphalt with penetration value between 40 and 50. However, the penetration value refers only to hardness of asphalt at room temperature. That makes this system lack of some information such as viscosity. The viscosity of asphalt should be measured because it helps to decide the mixing temperature and compaction temperature.

The viscosity grade is according to ASTM D3381 (ASTM International, 2018). The viscosity of asphalt is measured using a vacuum capillary viscometer. The number of viscosity grade relates to a viscosity value in the unit of poises at 60 °C. For example, AC-10 is the asphalt with its viscosity value of $1,000 \pm 200$ poises or AC-20 is the asphalt with its viscosity value of $2,000 \pm 400$ poises. However, these two grading systems do not provide overall important information such as the temperature range that the asphalt can be used. Therefore, the superpave performance grading system should be used, especially for polymer modified asphalt which is the special grade.



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ประวัติผู้เขียน

ชื่อ-สกุล	นพวรรณ คุณานุสรณ์
วัน เดือน ปี เกิด	-
สถานที่เกิด	-
วุฒิการศึกษา	-
ที่อยู่ปัจจุบัน	-

