CONVERSION OF CARBON DIOXIDE EMISSION USING CATALYTIC METHANATION METHOD IN HOT MIX ASPHALT

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DEDICATION

This thesis is dedicated to my mother (Fatimah Jusoh), my father (Abdul Rahman L. Wahab), all my supervisors, and all my teachers who taught me to always have a great faith, to keep working and never give up, because even the largest task can be accomplished if it is done one step at a time. I also dedicated this to my sisters (Faridah & Fazilah), brother (Mohd Yusof), my other family members, and all my best friends who always been beside me even during my hard time.

~ Thank you very much for always support me ~

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ABSTRACT

The purpose of this study is to mitigate the carbon dioxide (CO_2) emission from bitumen tank combustion unit in hot mix asphalt (HMA) plant. This study has been conducted by introducing the catalytic methanation method to reduce the CO_2 emission which majorly contributed to the greenhouse gases emissions in atmosphere. The benefit of using the method is that a high amount of CO_2 can be reduced without effecting the asphalt mixture properties which are very crucial to ensure high-quality asphalt pavement service life. This study suggested the conversion of CO_2 from flue gases emission to utilize it into methane (CH_4). The first stage of the study is the analysis of flue gases emissions from bitumen tank combustion unit in HMA plant by on-site gas analysis and laboratory analysis. The flue gas emission analysis shows that CO_2 is the major emission produced by combustion activities in bitumen tank combustion unit in HMA plant which the emission is between 4.95 - 15.55%. For the mitigation stage, Fourier Transform Infrared (FTIR) analysis is done to determine the percentage of CO_2 conversion and CH_4 formation over the catalyst used. After preparation and optimization, Ru/Sr/Ce (5:30:65)/Al_2O_3 catalyst calcined at 700°C for 5 hours and pre-treated at 300°C for 30 minutes with compressed air has been proposed as the best catalyst for the application of catalytic methanation method. This is because the catalyst produced the optimum values in term of CO_2 conversion and CH_4 formation during the reaction. The final stage of the study is the characterization of the catalyst to determine the factors contributed to its catalytic activities. The results show that the higher catalytic activities are caused by the uneven surface of catalyst with well shape hexagonal like particle on it. Besides that, the higher amount of Ruthenium (Ru) element composition in the catalyst, moderate basicity properties of the catalyst, and the higher pore volume in the catalyst also significantly contributed to its higher catalytic activities.

ABSTRAK

Tujuan kajian ini dilakukan adalah untuk mengurangkan pelepasan karbon dioksida (CO_2) dari unit pembakaran tangki bitumen pada loji asfalt campuran panas (HMA). Kajian ini telah dilakukan dengan memperkenalkan kaedah reaksi metanasi menggunakan pemangkin untuk mengurangkan pelepasan CO_2 yang banyak menyumbang kepada pelepasan gas hijau di atmosfera. Manfaat menggunakan kaedah ini adalah dapat mengurangkan jumlah CO_2 pada kadar yang lebih tinggi tanpa mengurangkan sifat mekanikal asphalt bagi memastikan jangka hayat turapan asfalt berkualiti tinggi dan tahan lama. Kajian ini mencadangkan penukaran CO_2 dari serombong untuk menghasilkan gas metana (CH_4). Peringkat pertama kajian ini adalah analisis pelepasan gas serombong dari unit pembakaran tangki bitumen dalam loji HMA menggunakan analisis gas secara langsung di loji HMA dan juga analisis di makmal. Analisis pelepasan gas serombong menunjukkan bahawa CO_2 adalah pelepasan utama yang disebabkan oleh aktiviti pembakaran dalam unit pembakaran tangki bitumen yang nilainya adalah antara 4.95 - 15.55%. Pada peringkat mitigasi, analisis *Fourier Transform Infrared* (FTIR) dilakukan untuk menilai peratusan penukaran CO_2 dan pembentukan CH_4 disebabkan oleh pemangkin yang digunakan. Selepas proses penyediaan dan pengoptimuman, pemangkin Ru/Sr/Ce (5:30:65)/Al_2O_3 yang dibakar pada suhu 700°C selama 5 jam dan dirawat pada suhu 300°C selama 30 minit menggunakan udara termampat telah dicadangkan sebagai pemangkin terbaik untuk kaedah mitigasi ini. Ini kerana pemangkin ini menghasilkan nilai peratusan optimum dari sudut penukaran CO_2 dan pembentukan CH_4 semasa reaksi. Seterusnya, tahap akhir kajian adalah pencirian pemangkin untuk menentukan faktor yang menyumbang kepada aktiviti pemangkin. Keputusan menunjukkan bahawa aktiviti reaksi metanasi yang lebih tinggi adalah disebabkan oleh permukaan yang tidak rata dan bentuk zarah heksagon yang terdapat pada permukaan pemangkin. Selain itu, kandungan Ruthenium (Ru) yang lebih tinggi dalam pemangkin, sifat bes sederhana pada pemangkin, dan juga jumlah liang yang lebih tinggi dalam pemangkin turut menyumbang kepada aktiviti metanasi pemangkin yang lebih baik dalam kaedah ini.

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LIST OF ABBREVIATIONS

CMA	-	Cold Mix Asphalt
CO_2-TPD	-	CO_2-Temperature Programmed Desorption
EDX	-	Energy Dispersion X-Ray
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infra-red
GHG	-	Greenhouse Gases
HMA	-	Hot Mix Asphalt
HR-TEM	-	High-Resolution Transmission Electron Microscopy
NA	-	Nitrogen Absorption
RSM	-	Response Surface Methodology
TEM	-	Transmission Electron Microscopy
UTM	-	Universiti Teknologi Malaysia
WMA	-	Warm Mix Asphalt
XRD	-	X-Ray Diffraction
XRF	-	X-Ray Fluorescent

LIST OF SYMBOLS

2θ	-	2 Theta
Al_2O_3	-	Aluminium Oxide
Å	-	Angstrom
Ce	-	Cerium
CH_4	-	Methane Gas
CO_2	-	Carbon Dioxide Gas
nm	-	Nanometer
ppm	-	Part per million
Ru	-	Ruthenium
Sn	-	Tin
Sr	-	Strontium
wt%	-	Weight percentage
Zr	-	Zirconium

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CHAPTER 1

INTRODUCTION

1.1 Hot Mix Asphalt (HMA) Plant Emissions

A hot mix asphalt (HMA) plant is an assembly of mechanical and electronic equipment in which aggregates, additives or sometimes recycled materials are blended, heated, dried and mixed together with binder (which usually bitumen) to produce asphalt mixture. The HMA plant may be stationary (located at a permanent location) or portable (moved from one site to another site) (INDOT, 2013).

In general, the common process in asphalt mixture production includes aggregate stacking, aggregate supply, bitumen heating, aggregate heating, and mixture mixing (Peng *et al.*, 2015). All emissions sources could produce flue gases emission such as carbon dioxide (CO_2), nitrogen oxides (NOx), hydrocarbons, carbon monoxide (CO), particulate matter and sulphur dioxide (SO_2), which are collectively known as greenhouse gases emissions (GHGs) (Arocho *et al.*, 2014).

1.2 Background of Study

The level of GHG emissions in HMA plants is tied to the development of road construction industry. The investment share of civil engineering work construction is 43.1% in 2018 out of overall value allocated for construction work in Malaysia. This means that about 3.9 billion USD has been allocated for this construction work, which includes the construction of roads, railways, and utilities (Department of Statistics Malaysia, 2019).

Malaysia has constructed over 200,000 km of roads, over 2,900 km of rail, 18 number of ports, and 22 number airports (Ambak *et al.*, 2014; Ministry of Transport Malaysia, 2018). The road network has increased by about 3.9 times from base year of 1995 (Ministry of Transport Malaysia, 2018). Hence, the rapid development of the road construction industry will significantly affected the environment as most of Malaysian used road as main transportation mode (Zakaria and Sufian, 2009). It is because, to cope with incremental industrial development, a road network must be constructed and maintained in good condition. Furthermore, the existing road also needs the regular maintenance through its service life to ensure the good quality of road for the users. HMA is one of the most important material for the road construction and maintenance.

Due to the high manufacturing temperature (150 to 190°C) and energy consumptions value of typical HMA mixtures, its production produced significantly high level of emissions (Ma *et al.*, 2019). The HMA production produced about 34000 kg CO_2-eq/km of road construction (Mazumder *et al.*, 2016). This value is based on the energy consumption equivalent value for the HMA production. Most of the CO_2 emissions from road construction was from the production of materials which included HMA production (Aurangzeb *et al.*, 2014; Pouranian and Shishehbor, 2019). For the HMA plant itself, about 34 to 38 kg CO_2 has been produced per 1 tonne of its production (Moretti *et al.*, 2017). Therefore, a way needs to be found to mitigate the emissions problem to maintain sustainability in terms of environmental conservation and to reduce the effects of global warming.

There is broad consensus that an excessive volume of greenhouse gases (GHGs) in the atmospheric system will have serious consequences in terms of climate change. Moreover, this could endanger human health, agricultural crops, forest species, various ecosystems and the overall environment as they enhance the greenhouse effect (Afroz *et al.*, 2003). A certain amount of GHGs exists in the atmospheric system and helps to absorb thermal radiation from the earth's surface, and then re-emits the radiation back to the earth, as shown in Figure 1.1.

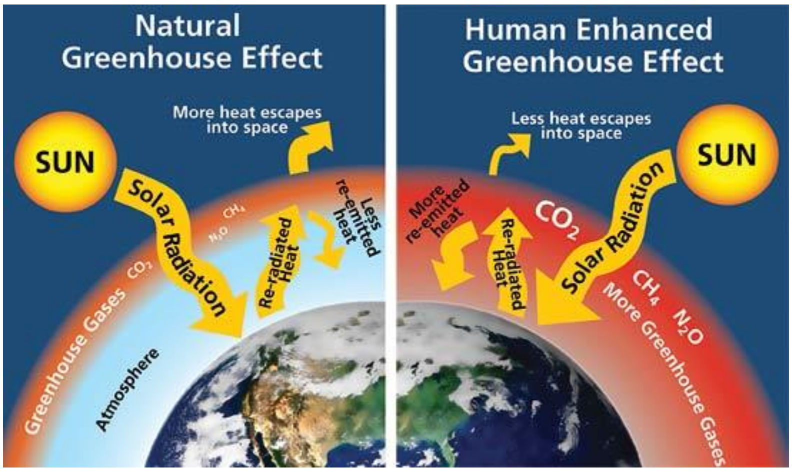

Figure 1.1 Greenhouse effect (Will Elder, 2018)

Figure 1.2 presents a comparison of the CO_2 emissions of Malaysia and several countries. From the figure, CO_2 emissions in Malaysia have increased significantly since 1985 (Theeyattuparampil *et al.*, 2013; The World Bank, 2014).

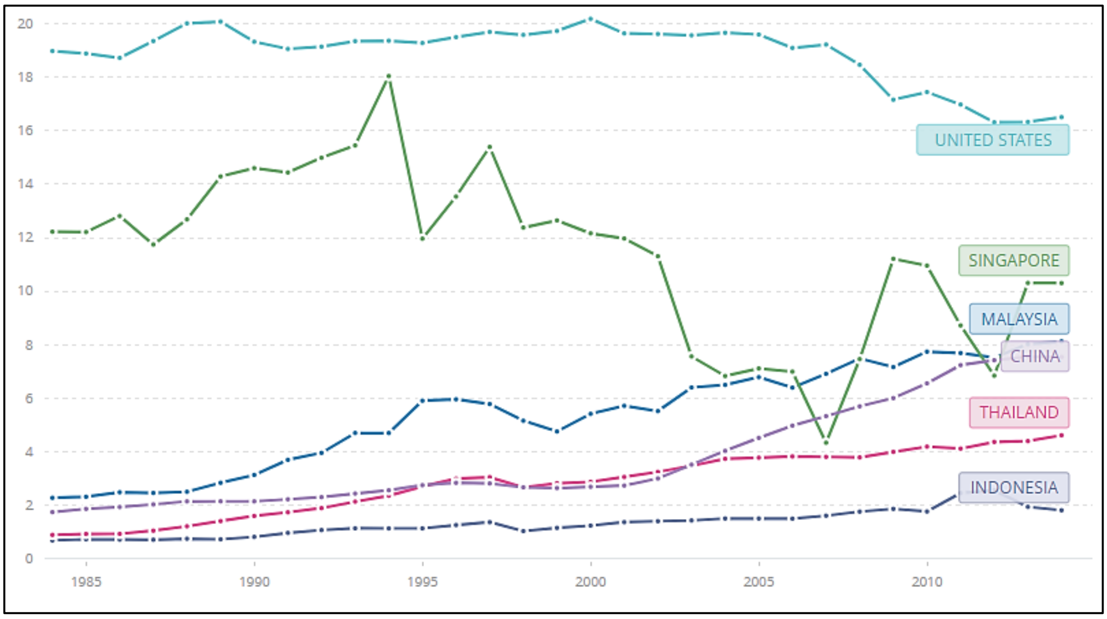

Figure 1.2 Carbon dioxide emissions (metric tons per capita) for Malaysia, Singapore, Indonesia, Thailand, United States, and China (The World Bank, 2014)

In addition , Malaysia cumulatively emitted 244.47 million tons of CO_2 at the end of 2019, with an annual growth rate of 0.4 percent and a share of 0.7 percent of global emissions in 2019 alone. (BP p.l.c., 2020). It is believed that these emissions will continue to increase in the future due to industrial development and economic growth.

In the past, the removal of CO_2 from the atmosphere occurred via photosynthesis, in which crops and other plants naturally consume CO_2 and sunlight and release oxygen (Arneth *et al.*, 2010) . However, due to recent rapid industrial development, plants alone are no longer able to deal with the amount of CO_2 in the atmosphere and remove it naturally (Peters *et al.*, 2012). Therefore, a sustainable solution must be found regarding this matter.

1.3 Problem Statement

Common mitigation of emissions from asphalt mixture production is the use of warm mix asphalt (WMA), cold mix asphalt (CMA), and modified HMA mixture to replace the traditional HMA during the asphalt material design. Reclaimed asphalt pavement (RAP) is also the common mitigation measure for the emission because it will prevent or reduce the emission of aggregates stacking, supply and heating process in HMA plant.

Almost all the available methods (WMA, CMA, and modified HMA) produced significantly good result in terms of emission mitigation for the asphalt mixture production. On the other hand, producing those asphalt materials at lower temperatures and while achieving the same high level of mechanical properties and field performance of asphalt mixture remains a challenge. Moreover, for RAP, the availability of material will make its application feasible. The details of the available methods reviewed are discussed in Section 2.1.

To fill the gap, this study has been conducted by introducing the catalytic methanation method to reduce the CO_2 emissions which majorly contributed to the

GHG emissions in atmosphere. The benefit of using the method is that a high amount of CO_2 can be reduced without effecting the asphalt mixer properties which are very crucial to ensure high-quality asphalt pavement service life. The method is the combination of carbon capture, sequestration, and utilize CO_2 to reduce the CO_2 emissions into the atmosphere and produce methane (CH_4) which is a source of fuel, hence mitigating GHG emissions to reduce global warming effects.

Methanation is cost effective technique. This process can be used to treat a large amount of CO_2 in a short time (Souma *et al.*, 2014). Furthermore, the catalysts are easy to prepare, environmentally friendly and reusable (Toemen, 2015). Moreover, the catalysts are safe to handle and only require minimum modifications to the existing systems in HMA plants, thus it not affecting the whole process of asphalt production in the plant.

1.4 Aim and Objectives of Research

The aim of this research is to mitigate CO_2 emissions from bitumen heating combustion unit during asphalt mix production process using the catalytic methanation method. This study suggested the conversion of CO_2 to utilize into CH_4 as mitigation method. In order to achieve this goal, several objectives have been set:

(a) To quantify the flue gases emission from bitumen heating combustion in HMA plant;

(b) To develop and optimize the novel catalyst for a catalytic methanation method; and

(c) To characterize the selected catalyst used for mitigation method.

1.5 Significant of Research

The importance of this research is it will capture the released emission from the chimney (post-combustion technique) without disturbing the original process of asphalt mixture production or bitumen heating in HMA plant. As a result, this approach will not reduce the mechanical properties of asphalt mixture during the material production process in HMA plant. Additionally, this will significantly mitigate GHG emissions and produce another low emission alternative energy (methane). The cycle of CO_2 conversion into another product is shown in Figure 1.4.

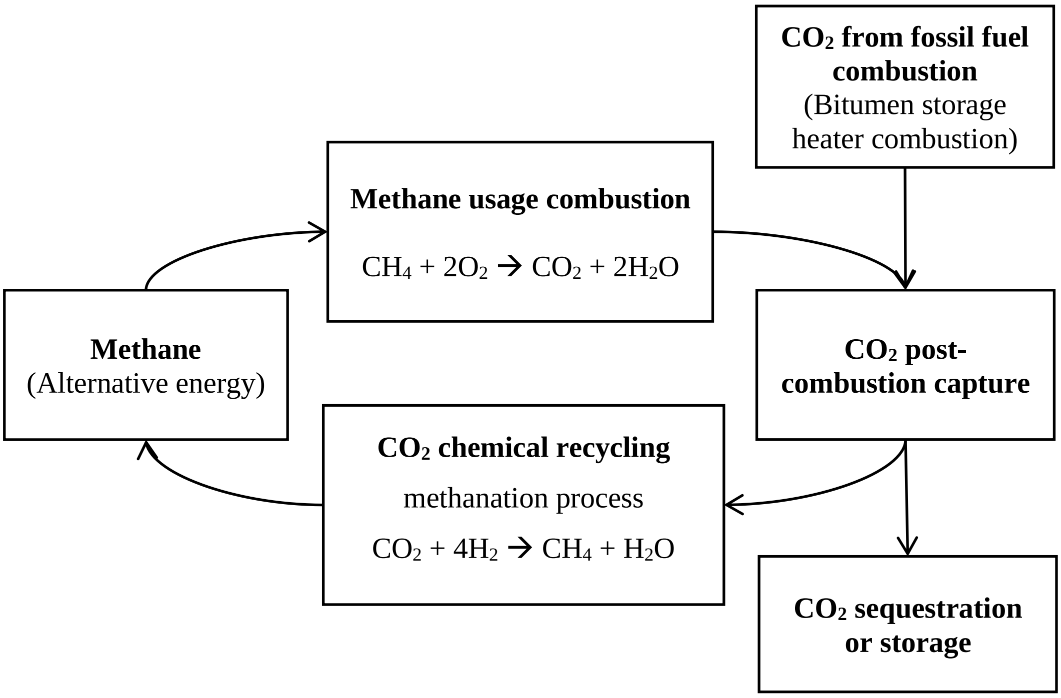

Figure 1.3 Carbon conversion cycle from CO_2 source to methane (Olah *et al.*, 2009)

The CO_2 was originally utilized from the product of fuel combustion. After the capture process, it is either sequestered or utilized for fuel feedstock to produce alternative fuel. The idea of this carbon cycle is adopted from Olah *et al.*, (2009) to be implemented in HMA plant.

In the past, the catalytic methanation technique has been used in various industries including power plant industry (Toemen, 2015; Thema *et al.*, 2019), oil and gas industry (Schaaf *et al.*, 2014), and cement industry (Baier *et al.*, 2018). However, no application of catalytic methanation has been used in HMA industry or to be specific on the combustion unit of bitumen tank in HMA plant.

The added value of this study is the method of quantifying the proportion of the emissions from the sources of combustion. This study quantified the emissions directly from the bitumen heating system's chimney. Then, the on-site measurement and laboratory analysis emission value is compared with the theoretically measured emission obtained from the form of fuel consumption.

1.6 Scope of Study

There are several scopes that limit the area of this study, which are:

(a) This study only intended to mitigate CO_2 emissions from bitumen heating combustion unit in an HMA plant.

(b) For the emissions quantification, the fuel used that are affected the flue gases emission is limited to light fuel oil only. Other types of fuel uses were not available for the visited HMA plants.

(c) The method of flue gases emission quantification is including theoretical method which adapted from United Nation Environment Program (UNEP, 2006), on-site gas analysis using portable gas analyser, and laboratory analysis by sampling the gas from HMA plant to the laboratory.

(d) The preparation method for the catalyst used in this study is incipient wetness impregnation method only. This method is adapted from the previous study conducted by Rosid, (2015); Toemen, (2015); and Sulaiman, (2016).

(e) The further catalyst optimization process in laboratory is done by referring to study conducted by Rosid, (2015); Toemen, (2015); Sulaiman, (2016); and Toemen *et al.*, (2016, 2018).

(f) For the statistical optimization, Response Surface Methodology (RSM) software is used in this study.

(g) The mitigation in this study covers up to the suitability or feasibility of the implementation of catalytic methanation at the bitumen heating combustion unit in HMA plant only. Further study needs to be done on the application of the system in a real site.

1.7 Thesis Outline

This study comprises seven chapters as follows:

Chapter 1 introduces the overall study which comprises the background, problem statement, objectives, significance of the study, and research scope.

Chapter 2 comprehensively discusses about the CO_2 mitigation method available from past studies. Besides that, it also discussed regarding the method introduced to mitigate the problem in this study. This chapter comprises a wide search of literature review, hence their merit as well as demerits are identified and discussed.

Chapter 3 presented the methods of the flue gases emission from HMA plant, preparation of materials for catalyst, a method to produce the catalyst, method of data analysis, and techniques to characterize the catalyst.

Chapter 4 presented the data obtained from the emission quantification of theoretical, on-site, and laboratory analysis from HMA plant. Furthermore, it presented the analysis results from all the method used.

Chapter 5 presented the data and analysis for the preparation and optimization of the catalyst. Then, the optimum catalyst is obtained from the laboratory as well as statistical optimization technique.

Chapter 6 presented the characterization technique for the catalyst. Furthermore, it discussed a detail of catalyst characteristic and presented the factor that affect the catalytic activities of catalyst.

Chapter 7 concludes the findings obtained from this study. Recommendations are also provided for potential future study.

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