# THE PERFORMANCE OF WARM MIX ASPHALT INCORPORATING ASPHALT BINDER IMPROVED WITH AMINE NANO-$TiO_2$ ADDITIVES

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# DEDICATION

**ALHAMDULILLAH**

Thank you to my beloved,

**FAMILY, LECTURERS & FRIENDS**

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## ABSTRACT

Warm mix asphalts (WMAs) are a green technology that requires low energy consumption and is more environmentally friendly than conventional hot mix asphalt (HMA) technologies. However, there are some drawbacks that cause WMAs to be less frequently adopted. As such, further research is warranted. To that end, this study examined the possibility of modifying asphalt binders with nano-$TiO_2$ (titanium dioxide nanoparticles) for use in WMAs. Initially, a PG70 asphalt binder was modified with 9.0% nano-$TiO_2$ to meet the performance requirements of a PG76 asphalt binder. As the viscosity of the 9% nano-$TiO_2$ modified asphalt binder was marginally higher than that of unmodified PG70 asphalt binders, the 9.0% nano-$TiO_2$ was further modified by its total weight with 5.0%, 5.5%, 6.0%, and 6.5% octadecylamine (OCTA) and 3.5%, 4.0%, 4.5%, and 5.0% 3-aminopropyltriethoxysilane (TRI) to produce new nanoparticle additives namely amine nano-$TiO_2$ OCTA (TIOCTA1, TIOCTA2, TIOCTA3, and TIOCTA4) and amine nano-$TiO_2$ TRI (TIOTRI1, TIOTRI2, TIOTRI3, and TIOTRI4). These new nanoparticle additives were then added to PG70 asphalt binders to improve the workability of asphalt mixtures at lower temperatures. After mixed, the asphalt binders were then evaluated in terms of storage stability as well as physical, rheological, and morphological characteristics. Then, the asphalt mixtures incorporating these asphalt binders were compacted using temperatures that were 20°C to 40°C lower than that required by HMA to produce WMAs. The performance of all samples was evaluated in terms of resistance to rutting, fatigue, and moisture-induced damage via a resilient modulus test, indirect tensile fatigue (ITF) test, dynamic creep (DC) test, and a modified Lottman test. The test results indicated that the performance of the modified asphalt binders had improved to PG76 asphalt binder, homogeneous, stable in storage, and had a high modulus. Furthermore, all TIOCTA and TIOTRI asphalt mixtures that had been produced at lower temperatures

were more resistant to fatigue, rutting, and moisture-induced damage than those of HMA. A comparative study also concluded that the TIOTRI3 asphalt mixture compacted at 30°C lower than the HMA, had significantly better resistance to rutting, fatigue and moisture-induced damage.

**ABSTRAK**

Asfalt campuran suam (WMA) merupakan teknologi hijau yang memerlukan penggunaan tenaga yang rendah dan mesra alam sekitar berbanding teknologi asfalt campuran panas (HMA). Namun begitu, terdapat beberapa kelemahan pada teknologi WMA yang menyebabkan ia tidak digunakan secara meluas. Oleh itu penyelidikan lanjut adalah diperlukan. Kajian ini dijalankan untuk menilai potensi asfalt pengikat yang mengandungi nano-$TiO_2$ (partikel nano titanium dioxida) untuk digunakan di dalam WMA. Pada mulanya, asfalt pengikat asli gred PG70 diubahsuai dengan 9.0% nano-$TiO_2$ untuk menghasilkan asfalt pengikat gred PG76. Memandangkan asfalt pengikat terubahsuai 9.0% nano-$TiO_2$ mempunyai kelikatan yang ketara lebih tinggi dari asfalt pengikat asli, partikel 9.0% nano-$TiO_2$ kemudian telah diubahsuai dengan 5.0%, 5.5%, 6.0% dan 6.5% Octadecylamine (OCTA); dan 3.5%, 4.0%, 4.5% dan 5.0% 3-Aminopropyltriethoxysilane (TRI) dari berat keseluruhannya bagi menghasilkan bahan tambah nanopartikel baharu dinamakan sebagai Amina nano-$TiO_2$ OCTA (TIOCTA1, TIOCTA2, TIOCTA3 dan TIOCTA4) Amina nano-$TiO_2$ TRI (TIOTRI1, TIOTRI2, TIOTRI3 dan TIOTRI4). Bahan-bahan tambah nanopartikel baharu ini kemudian dicampurkan dengan asfalt pengikat asli untuk memperbaiki kebolehkerjaan campuran asfaltnya pada suhu yang lebih rendah. Setelah dicampurkan, semua asfalt pengikat tersebut telah dinilai berdasarkan ciri-ciri kestabilan dalam simpanan, fizikal, rheologi dan morfologi. Kemudian, campuran asfalt yang mengandungi setiap asfalt pengikat terubahsuai dipadatkan antara 20°C hingga 40°C lebih rendah dari HMA untuk menghasilkan WMA. Seterusnya, semua campuran asfalt diuji kebolehtahanan terhadap pesongan, kelesuan dan kegagalan lembapan dengan menggunakan ujian modulus keanjalan, ujian keterikan pesongan tidak langsung, ujian rayapan dan ujian Lottman

terubahsuai. Keputusan bagi ujian-ujian yang dijalankan menunjukkan bahawa asfalt pengikat terubahsuai telah mencapai gred PG76, homogen, stabil dalam simpanan dan mempunyai moduli yang tinggi. Tambahan lagi, semua campuran asfalt terubahsuai yang dihasilkan menggunakan suhu yang rendah mempunyai kebolehtahanan yang tinggi terhadap kelesuan, pesongan dan kegagalan lembapan berbanding HMA. Daripada perbandingan yang dibuat, campuran TIOTRI3 yang dimampatkan pada suhu 30°C lebih rendah dari HMA adalah bertambah baik dengan signifikan dari segi ketahanan terhadap pesongan, kelesuan dan kegagalan lembapan.

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## LIST OF SYMBOLS AND ABBREVIATIONS

| | | |
|---|---|---|
| $\alpha$ | - | Level of significance |
| $\delta$ | - | Phase Angle |
| $\varepsilon_{3600}$ | - | Denotes the strain at $3600^{th}$ cycle |
| $\varepsilon_{1200}$ | - | Denotes the strain at $1200^{th}$ cycle |
| $p$ | - | Probability Value |
| $\eta$ | - | Viscosity |
| $\|E^*\|$ | - | Dynamic Modulus |
| *F*-ratio | - | Ratio Between-Group Variance and Within-Group Variance |
| $G_{mm}$ | - | Maximum Specific Gravity |
| $G^*$ | - | Complex Modulus |
| $G^*/\sin\delta$ | - | Rutting Parameter for Asphalt Binder |
| $G^*\sin\delta$ | - | Fatigue Parameter for Asphalt Binder |
| $H_o$ | - | Null Hypothesis |
| $SO_2$ | - | Sulphur Dioxide |
| $R^2$ | - | Coefficient of Determination |
| $CaCO_3$ | - | Calcium Carbonat |
| $CO_2$ | - | Carbon Dioxide |
| CO | - | Carbon Oxide |
| CM | - | Carbon microfiber |
| $NO_x$ | - | Nitrogen Oxide |
| $N_2$ | - | Nitrogen Gas |
| $M_R$ | - | Resilient Modulus |
| $Fe_3O_4$ | - | Ferro Phosphorus |

| | | |
|---|---|---|
| EEIP | - | Energy Efficiency in Industrial Processes |
| WMA | - | Warm Mix Asphalt |
| HMA | - | Hot Mix Asphalt |
| PMA | - | Polymer modified asphalt |
| IDT | - | Indirect Tensile Test |
| ITS | - | Indirect Tensile Strength |
| LVDT | - | Linear Variable Displacement Transducers |
| Nano-$TiO_2$ | - | Nano-Titanium Oxide |
| NMAS | - | Nominal Maximum Aggregate Size |
| ANOVA | - | One-way Analysis of Variance |
| OBC | - | Optimum Binder Content |
| PAV | - | Pressure Aging Vessel |
| PI | - | Penetration Index |
| PG | - | Performance Grading |
| PVN | - | Penetration Viscosity Number |
| Ra | - | Surface Roughness |
| RTFO | - | Rolling Thin Film Oven |
| RV | - | Rotational Viscometer |
| SEM | - | Scanning Electron Microscopy |
| SEM-EDS | - | Scanning Electron Microscopy/ Energy Dispersive X-Ray Spectroscopy |
| SFE | - | Surface Free Energy |
| SHRP | - | Strategic Highway Research Program |
| SPSS | - | Statistical Package for the Social Sciences |
| TSR | - | Tensile Strength Ratio |
| FESEM | - | Field Emission Scanning Electron Microscopy |
| CSS | - | Creep Strain Slope |
| DSR | - | Dynamic Shear Rheometer |
| XRD | - | X-Ray diffraction |
| FTIR | - | Fourier Transform Infrared |
| UTM | - | Universal Testing Machine |
| UV | - | Ultra Violet |
| VFA | - | Void Filled with Asphalt |

| | | |
|---|---|---|
| VTM | - | Void in Total Mixture |
| AV | - | Air Void |
| XRD | - | X-ray Diffraction |
| OCTA | - | Octadecylamine |
| TRI | - | 3-Aminopropyltriethoxysilane |
| MARC | - | Modified Asphalt Research Centre |
| NM | - | Nanomer material |
| NS | - | Nano-silica |
| NNC | - | Non-modified nano-clay |
| PNC | - | Polymer modified nano-clay |
| ASTM | - | American Standard Testing Method |
| AASHTO | - | American Association of State Highway and Transportation Officials |
| MS | - | Malaysian Standard |
| EPA | - | Environment Protection Agency |
| RTFOT | - | Rolling Thin Film Oven Test |
| DOT | - | Department of Transportation's |
| HCl | - | Hydro Chloric acid |
| TBOT | - | Titanium (IV) butoxide |
| CTAB | - | Cetyl Trimethylammonium bromide |
| NaOH | - | Sodium hydroxide |
| STA | - | Short term ageing |
| LTA | - | Long term ageing |
| BET | - | Brunauer-Emmett-Teller |
| UTHM | - | Universiti Tun Hussein Onn Malaysia |
| MiNT-SRC | - | Microelectronics & Nanotechnology - Shamsuddin Research Centre |
| MRB | - | Malaysian Rubber Board |
| JKR | - | Public work department |
| TIO2P | - | Type1 nano-$TiO_2$ particle |
| TIO2F | - | Type2 nano-$TiO_2$ particle |
| TIO2R | - | Type3 nano-$TiO_2$ particle |
| TIO2P5 | - | TIO2P asphalt binder + 5.0% TIO2P |
| TIO2P7 | - | TIO2P asphalt binder + 7.0% TIO2P |

| | | |
|---|---|---|
| TIO2P9 | - | TIO2P asphalt binder + 9.0% TIO2P |
| TIOCTA1 | - | TIO2P9 asphalt binder + 5.0% TIOCTA1 |
| TIOCTA2 | - | TIO2P9 asphalt binder + 5.5% TIOCTA2 |
| TIOCTA3 | - | TIO2P9 asphalt binder + 6.0% TIOCTA3 |
| TIOCTA4 | - | TIO2P9 asphalt binder + 6.5% TIOCTA4 |
| TIOTRI1 | - | TIO2P9 asphalt binder + 3.5% TIOTRI1 |
| TIOTRI2 | - | TIO2P9 asphalt binder + 4.0% TIOTRI2 |
| TIOTRI3 | - | TIO2P9 asphalt binder + 4.5% TIOTRI3 |
| TIOTRI4 | - | TIO2P9 asphalt binder + 5.0% TIOTRI4 |

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# CHAPTER 1

# INTRODUCTION

## 1.1 Background of the research

The design and construction of sustainable infrastructure has become a burgeoning trend of the 21$^{st}$ century (Nuramo & Haupt, 2017; Uchehara *et al*., 2022; Mansur *et al*., 2022). As such, recent efforts to develop sustainable road infrastructure focus on reducing energy consumption, minimising emissions, practising cleaner manufacturing processes, and utilising recycled materials in asphalt mixture design (Newman *et al*., 2012; Zhang, 2018; Nwakaire *et al.* 2020; Uchehara *et al*., 2022).

Conventional asphalt mixtures; specifically, hot mix asphalts (HMAs); are produced at temperatures ranging between 140°C to 190°C, require high energy consumption for heating, and have high environmental emissions (Abdullah *et al*., 2014; Sol-Sánchez *et al*., 2018; Yao *et al*., 2019; Xiao *et al*., 2019; Xiu *et al.*, 2020; Sukhija *et al*., 2021). Furthermore, as the binders used in these asphalt mixtures are viscoelastic, they are extremely temperature sensitive. As such, the high temperatures that it is subjected to during mixture production accelerates its ageing, resulting in a difficult compaction process (Arega & Bhasin, 2012). These high temperatures also cause rutting damage (Bairgi *et al*., 2018). Furthermore, prolonged service periods also accelerate the formation of cracks on the surface of the asphalt pavement due to oxidation and hardening. This damage becomes more critical when the asphalt pavement is subjected to heavy annual precipitation, prolonged rainfall, and increased ground water levels or flooding. Under these conditions, the stiffness of the asphalt pavement reduces, resulting in moisture damage; such as stripping and potholes.

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**APPENDIX H**

## LIST OF AWARD AND PUBLICATIONS

### 1. Award

(i) Bronze Medal Awarded, Sustainable Asphaltic Concrete Incorporated Hydrothermal Nano-Titanium Dioxide, *2nd FKAAS Innovation Festival 2017* (*InnoFest'17*). 2017.

### 2. Proceedings

(i) Buhari, R., Abdullah, M. E., Ahmad, M. K., Tajudin, S. A. and Abu Bakar, S. K. Laboratory Study on The Fatigue Resistance of Asphaltic Concrete Containing Titanium Dioxide. *E3S Web of Conferences*. 2018. 3:01021.

(ii) Buhari, R., Abdullah, M. E., Ahmad, Haini, R., Abu Bakar, S. K. Physical and Rheological Properties of Titanium Dioxide Modified Asphalt. *E3S Web of Conferences*. 2018. 34: 01035.

### 3. Journals

(i) Buhari, R., Abdullah, M. E., Ahmad, M. K., Zabidi, N. and Bakar, S. K. A. Moisture Susceptibility of Modified Asphalt Concrete Containing Titanium Dioxide, *International Journal of Advanced Trends in Computer Science and Engineering*. 2019. 8:140-143.

(ii) Buhari, R., Abdullah, M. E., Ahmad, M. K. Hainin, R. and Bakar, S. K. A. Fatigue Performance of Titanium Dioxide Modified Asphaltic Concrete, *International Journal of Advanced Trends in Computer Science and Engineering*. 2019. 8:131-135.

(iii) Buhari, R., Abdullah, M. E., Ahmad, M. K., Tajudin, S. A. and Abu Bakar, S. K. Laboratory Study on The Fatigue Resistance of Asphaltic Concrete Containing Titanium Dioxide, *E3S Web of Conferences*. 2018. 34:01021.