

RHEOLOGICAL AND VISCOELASTIC PROPERTIES OF ASPHALT BINDER MODIFIED
WITH NANO-SILICA AND SASOBIT

by

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Under the supervision of

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DEDICATION

Dedicated to my parents

CERTIFICATE

This is to certify that the thesis titled “**RHEOLOGICAL AND VISCOELASTIC PROPERTIES OF ASPHALT BINDER MODIFIED WITH NANO-SILICA AND SASOBIT**” by Karanjeet Kaur to the Indian Institute of Technology Delhi, for the award of the degree of Doctor of Philosophy, is a bona fide record of the research work done by him under my supervision. The contents of this thesis, in full or in parts, have not been submitted to any other Institute or University for the award of any degree or diploma.

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ABSTRACT

RHEOLOGICAL AND VISCOELASTIC PROPERTIES OF ASPHALT BINDER MODIFIED WITH NANO-SILICA AND SASOBIT

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Load on pavements has increased due to the intensive number of vehicles. The life of pavements has been abbreviated due to this rapid overloading in terms of distresses like permanent deformation and fatigue failure, which leads to continued maintenance. Bitumen should have lower stiffness at lower temperatures and higher stiffness at a higher temperature for satisfactory performance under various distresses. It should not be brittle at lower temperatures, causing fatigue failure or thermal cracking. Neat bitumen behaves like liquid at higher temperatures (due to lower viscosity), leading to permanent deformation. Neat bitumen has some limitations under specific requirements, which can be accomplished using additives in bitumen.

Warm Mix Asphalt can decrease construction expenses and environmental effects by reducing fuel consumption and emissions. Moreover, various WMA additives or processes can enhance multiple rheological properties of modified asphalt. However, it has some issues due to reduced manufacturing temperatures, like moisture damage due to partial dry aggregate and rutting due to lower aging. Aliphatic polymethylene hydrocarbon (typically long chain) produced from coal gasification using the Fischer-Tropsch (FT) process is helpful in reduction of emission, production temperatures, and

fuel consumption. Even though, Sasobit has been extensively used in this research, the thesis does not promote any particular commercial product.

Nanomaterials are gaining importance due to their massive explicit surface area and superior dispersion ability. Various nanomaterials have improved the performance of the modified asphalt. Nano-Silica has improved the anti-aging performance due to high surface area, large pore size, and hydroxyl groups. The oxygen atom present in hydroxyl groups forms hydrogen bonds with aromatic sheets present in the polar aromatics and asphaltene molecules in asphalt. In this interaction, the oxygen affinity is reduced, and oxidation is declined. The hydroxyl groups also help to promote hydrogen bonding with fused aromatic rings to develop the barrier for oxidation.

In this study, the effect of Sasobit (WMA additive) and Nano-Silica (Nanomaterial) and their interaction on Viscosity Grade (VG)10 and 30 binders have been investigated. The study is conducted in three parts. Part one is the characterization of Sasobit modified binders, and part two is the characterization of Nano-Silica modified binders. Part three explains the characterization of Sasobit and Nano-Silica interaction. For part one, modified binders were prepared with four dosages of Sasobit with both VG10 and VG30. For part two, modified binders were prepared with three dosages of Nano-Silica with both VG10 and VG30. For part three, modified binders were prepared with the interaction of Sasobit and Nano-Silica with both VG10 and VG30. For part four, optimum dosages were determined from the test results obtained from objectives one, two, and three, respectively, using Response Surface Methodology (RSM). Total one ninety-eight blends were prepared under different aging conditions to understand the

aging effect of modified binders. These unaged blends were aged for short-term, long-term, and extended aging conditions.

Total of seven aging conditions were used to study the aged effect. Total 70 blends of Sasobit modified binder were tested to evaluate the rheological and viscoelastic properties. Brookfield rotational viscometer was used to measure the viscosity from 100 °C to 180 °C. Fourier transform infrared spectroscopy was used to assess quantifiable variations with chemical bonding and chemical structure. Frequency sweep test was used to measure the viscoelastic properties for unaged, short-term aged, long-term aged and extended aging conditions at a frequency range of 50 Hz to 0.01 Hz from 10°C to 90°C at 10°C interval. Nano-Silica was used in three dosages for part two of the study. Standard and modified storage stability tests were conducted to evaluate storage stability. For part three, the interaction of both additives was used. The methodology of part one was adopted for parts two and three. The optimum dosage of additives was determined using Response Surface Methodology.

Various parameters were used to measure intermediate and high-temperature performance, such as conventional properties, fatigue parameter, rutting parameter, zero shear viscosity, non-recoverable compliance, and activation energy. It was observed by conventional tests that stiffness was increased by Sasobit, and Nano-Silica modified asphalt binders. At higher dosages of Sasobit, elasticity was reduced at lower temperatures. Sasobit addition reduced the viscosity by improving the workability for lower production temperatures. However, increase in viscosity was found by increasing Nano-Silica content. In general, complex modulus increase and decrease in phase angle was found with an increase in Sasobit content and Nano-Silica content at all aging

conditions. Activation energy was decreased by increasing Sasobit (up to certain dosages) and Nano-Silica content.

Limiting temperatures, rutting parameter and zero shear viscosity were increased with increase in Sasobit and Nano-Silica increase. Lower Jnr values showed the improved rutting resistance. Thus, it can be concluded that Sasobit and Nano-Silica improved the high temperature performance of modified asphalt. Nano-Silica also improved the anti-aging property of Sasobit modified asphalt binders due to lower aging indexes.

Disclaimer statement: The additives, Sasobit and Nano-Silica used in this study do not promote any company. The similar names have been used in other studies too.

सारांश

वाहनों की अधिक संख्या के कारण सड़क पर दबाव बढ़ गया है। तेजी से बढ़ रहे अधिक भारी वाहनों के संकट के कारण स्थायी विकृति और फैटीग फेलियर के रूप में सड़क का जीवन संक्षिप्त होता जा रहा है जिसके कारण सड़क की लगातार मरम्मत आवश्यक है ऐसे विभिन्न संकटों में संतोषजनक प्रदर्शन के लिए बिटुमेन में कम तापमान पर कम कठोरता और उच्च तापमान पर उच्च कठोरता होनी चाहिए। यह कम तापमान पर भंगुर नहीं होना चाहिए, जिससे फैटीग फेलियर या उष्मीय दरारें उत्पन्न हो। साफ बिटुमेन उच्च तापमान (कम श्यानता के कारण) पर तरल की तरह व्यवहार करता है, जिससे स्थायी विरूपण होता है। विशिष्ट आवश्यकताओं के तहत स्वच्छ बिटुमेन की कुछ सीमाएँ होती हैं, जिन्हें बिटुमेन में एडिटिक्स का उपयोग करके पूरा किया जा सकता है।

गरम मिश्रित डामर, ईंधन की खपत और उत्सर्जन को कम करके निर्माण खर्च और पर्यावरणीय प्रभावों को कम कर सकता है। इसके अलावा, विभिन्न डब्ल्यू.एम्.ए. एडिटिक्स या प्रक्रियाएं, संशोधित डामर के कई रियोलॉजिकल गुणों को बढ़ा सकती हैं। हालांकि, कम विनिर्माण तापमान के कारण इसमें कुछ समस्याएं हैं, जैसे आंशिक शुष्क अग्रग्रेट्स के कारण नमी की क्षति और कम एजिंग के कारण रट्टिंग। फिशर-ट्रॉप्स (एफटी) प्रक्रिया का उपयोग करके कोयला गैसीकरण से उत्पादित एलीफैटिक पॉलीमेथिलीन हाइड्रोकार्बन (आमतौर पर लंबी श्रृंखला) उत्सर्जन, उत्पादन तापमान और ईंधन की खपत को कम करने में सहायक है। यद्यपि इस शोध में सैसोबिट का व्यापक रूप से उपयोग किया गया है, तथापि शोध किसी विशेष व्यावसायिक उत्पाद को बढ़ावा नहीं देता है।

नैनोमटेरियल्स अपने विस्तृत स्पष्ट सतह क्षेत्र और बेहतर फैलाव क्षमता के कारण महत्वपूर्ण हैं। विभिन्न नैनो सामग्री ने संशोधित डामर के प्रदर्शन में सुधार किया है। नैनो-सिलिका ने वृहद सतह क्षेत्र, बड़े छिद्रों के आकार और हाइड्रॉक्सिल समूहों के कारण एंटी-एजिंग प्रदर्शन में सुधार किया है। हाइड्रॉक्सिल समूहों में मौजूद ऑक्सीजन परमाणु, ध्रुवीय एरोमेटिक्स में मौजूद एरोमेटिक सतहों के साथ हाइड्रोजन बंधन और डामर में अश्फाल्टीन अणुओं का निर्माण करता है। इस क्रिया में,

ऑक्सीजन आकर्षण कम हो जाता है, और ऑक्सीकरण कम हो जाता है। हाइड्रॉक्सिल समूह ऑक्सीकरण के लिए अवरोध विकसित करने के लिए जुड़े हुए एरोमेटिक्स रिंग्स के साथ हाइड्रोजन बंधन को बढ़ावा देने में भी मदद करते हैं।

इस अध्ययन में, सैसोबिट (डब्ल्यू.एम्.ए.एडिटिव) और नैनो-सिलिका (नैनोमेटेरियल) के प्रभाव और श्यानता श्रेणी (वीजी) 10 और 30 बाइंडर्स पर उनकी परस्पर-क्रिया की जांच की गई है। अध्ययन तीन भागों में किया गया है। भाग एक सैसोबिट संशोधित बाइंडर्स का लक्षण वर्णन है, और भाग दो नैनो-सिलिका संशोधित बाइंडर्स का लक्षण वर्णन है। भाग तीन सासोबिट और नैनो-सिलिका परस्पर-क्रिया के लक्षण वर्णन की व्याख्या करता है। इस अध्ययन के उप उद्देश्य एवं तदनुरूपी परिणाम निम्नानुसार हैं।

भाग एक के लिए, सैसोबिट को चार अलग अलग स्तर पर VG10 और VG30 दोनों के साथ मिश्रित कर संशोधित बाइंडर्स तैयार किए गए। भाग दो के लिए, VG10 और VG30 दोनों के साथ नैनो-सिलिका का तीन स्तर पर मिश्रण कर संशोधित बाइंडर तैयार किए गए। भाग तीन के लिए, संशोधित बाइंडर्स, VG10 और VG30 दोनों के साथ सैसोबिट और नैनो-सिलिका के परस्पर मिश्रण के साथ तैयार किए गए। भाग चार के लिए, रिस्पांस सरफेस मथडोलॉजी (RSM) का उपयोग करते हुए, क्रमशः एक, दो और तीन उद्देश्यों से प्राप्त परीक्षण परिणामों से इष्टतम मात्रा निर्धारित की गई। संशोधित बाइंडर्स के एजिंग बढ़ने के प्रभाव को समझने के लिए विभिन्न एजिंग स्थितियों वाले कुल एक सौ अठानबे मिश्रण तैयार किए गए। इन मिश्रणों को अल्पकालिक, दीर्घकालिक और विस्तारित एजिंग परिस्थितियों के अनुसार ऐज किया गया।

एजिंग स्थितियों का अध्ययन करने के लिए कुल सात एजिंग परिस्थितियों का उपयोग किया गया। सैसोबिट संशोधित बाइंडर के कुल 70 मिश्रणों का परीक्षण रियोलॉजिकल और विस्कॉइलास्टिक गुणों का मूल्यांकन करने के लिए किया गया। ब्रुकफील्ड घूर्णी विस्कोमीटर का उपयोग श्यानता को 100 डिग्री सेल्सियस से 180 डिग्री सेल्सियस तक मापने के लिए किया गया। फूरियर ट्रांसफॉर्म इंफ्रारेड स्पेक्ट्रोस्कोपी का उपयोग रासायनिक बंधन और रासायनिक संरचना के साथ मात्रात्मक भिन्नताओं का आकलन करने के लिए किया गया। फ्रीक्वेंसी स्वीप टेस्ट का उपयोग 10°C से 90°C के अंतराल पर 50 हर्ट्ज से 0.01 हर्ट्ज की आवृत्ति रेंज में बिना ऐज किए हुए, अल्पकालिक ऐज, दीर्घकालिक ऐज और

विस्तारित एज स्थिति के लिए विस्कोलेस्टिक गुणों को मापने के लिए किया गया। अध्ययन के भाग दो के लिए नैनो-सिलिका का उपयोग तीन मिश्रण स्तरों में किया गया। स्टोरेज स्टैबिलिटी का मूल्यांकन करने के लिए मानक और संशोधित स्टोरेज स्टैबिलिटी परीक्षण किए गए। भाग तीन के लिए, दोनों एडिटिव्स की परस्पर क्रिया का उपयोग किया गया। भाग एक की पद्धति को भाग दो और तीन के लिए अपनाया गया। एडिटिव्स की इष्टतम मात्रा रिस्पांस सरफेस मेथडोलॉजी का उपयोग करके निर्धारित की गई।

पारंपरिक गुणों, फैटीग पैरामीटर, रट्टिंग पैरामीटर, शून्य शिफ्ट श्यानता, नॉन-रिकवरएबल कंप्लायंस, और सक्रियण ऊर्जा जैसे मध्यवर्ती और उच्च तापमान प्रदर्शन को मापने के लिए विभिन्न मानकों का उपयोग किया गया था। यह पारंपरिक परीक्षणों द्वारा देखा गया था कि सैसोबिट और नैनो-सिलिका द्वारा संशोधित बाइंडर्स की कठोरता में वृद्धि की गई। सैसोबिट की उच्च प्रतिशत पर, कम तापमान पर इलास्टिसिटी कम हो गई।

सैसोबिट द्वारा कम उत्पादन तापमान पर श्यानता को कम करते हुए वर्कएबिलिटी में सुधार किया गया। हालांकि, नैनो-सिलिका सामग्री में वृद्धि से श्यानता में वृद्धि पाई गई। सामान्य तौर पर, सभी एजिंग स्थितियों में सैसोबिट मात्रा और नैनो-सिलिका मात्रा में वृद्धि के साथ काम्प्लेक्स मॉड्युलस वृद्धि और फेज एंगल में कमी पाई गई। सैसोबिट (कुछ मात्रा तक) और नैनो-सिलिका सामग्री को बढ़ाने से सक्रियण ऊर्जा को में कमी हुई। सैसोबिट मात्रा और नैनो-सिलिका मात्रा में वृद्धि के साथ लिमिटिंग तापमान, रट्टिंग पैरामीटर और शून्य शिफ्ट श्यानता में वृद्धि हुई। कम JNR पर बेहतर रट्टिंग प्रतिरोध प्रदर्शित हुआ। इस प्रकार, यह निष्कर्ष निकाला जा सकता है कि सैसोबिट और नैनो-सिलिका ने संशोधित डामर के उच्च तापमान प्रदर्शन में सुधार किया। नैनो-सिलिका के न्यून एजिंग सूचकांक के कारण सैसोबिट संशोधित डामर बाइंडर्स की एंटी-एजिंग गुण में भी सुधार किया।

अस्वीकरण कथन:- इस अध्ययन में इस्तेमाल किए गए एडिटिव्स, सैसोबिट और नैनो-सिलिका किसी भी कंपनी का प्रचार नहीं करते हैं। इसी तरह के नामों का इस्तेमाल अन्य अध्ययनों में भी किया गया है।

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LIST OF ABBREVIATIONS AND SYMBOLS

<i>AI</i>	Aging Index
<i>AFM</i>	Atomic Force Microscopy
<i>APA</i>	Asphalt Pavement Analyzer
<i>BBR</i>	Bending Beam Rheometer
<i>BOS</i>	Bond Strength Ratio
<i>CMA</i>	Cold Mix Asphalt
<i>CSV</i>	Compressive Strength Value
<i>CS</i>	Creep Stiffness
<i>DCT</i>	Dynamic Creep Test
<i>DSR</i>	Dynamic Shear Rheometer
<i>ESO</i>	Epoxidized Soybean Oil
<i>GPC</i>	Gel Permeation Chromatography
<i>HWT</i>	Hamburg Wheel Tracking
<i>HMA</i>	Hot Mix Asphalt
<i>HWMA</i>	Half Warm Mix Asphalt
<i>FTIR</i>	Fourier Transform Infrared Spectroscopy
<i>FPBFT</i>	Four Point Bending Fatigue Test
<i>IDT</i>	Indirect Tensile Strength
<i>IR</i>	Infrared Spectrum
<i>I_{Ar}</i>	Aromatic index
<i>I_{Al}</i>	Aliphatic index

<i>I_{C=O}</i>	Carbonyl index
<i>I_{S=O}</i>	Sulphoxide index
<i>ITFT</i>	Indirect Tensile Fatigue Test
<i>LAS</i>	Linear Amplitude Sweep
<i>MSCR</i>	Multiple Stress Creep Recovery
<i>MS</i>	Marshall Stability
<i>PAV</i>	Pressure Aging Vessel
<i>P20</i>	Pressure Aging Vessel 20 hours
<i>POTS</i>	Pull-Off Tensile Strength Ratio
<i>UA</i>	Unaged
<i>VG</i>	Viscosity Grade
<i>FS</i>	Flexural Strength
<i>RMS</i>	Retained Marshall stability
<i>RTFO</i>	Rotational Thin Film Oven
<i>R85</i>	Rotational Thin Film Oven (85 minutes)
<i>RR</i>	Rate Of Rutting
<i>RD</i>	Rut Depth
<i>RV</i>	Rotational Viscosity
<i>RM</i>	Resilient Modulus
<i>RS</i>	Recoverable Strain
<i>SBR</i>	Styrene-butadiene Rubber
<i>SSS</i>	Standard Storage Stability
<i>SSM</i>	Modified Storage Stability

<i>TFOT</i>	Thin Film Oven Test
<i>TSR</i>	Tensile Strength Ratio
<i>TTSP</i>	Time Temperature Superposition Principle
<i>VTS</i>	Viscosity temperature susceptibility
<i>WMA</i>	Warm Mix Asphalt
<i>WB</i>	Water Surfactant Based
<i>WWT</i>	Wessex Wheel Tracking
<i>WTT</i>	Wheel Tracking Test
<i>WTA</i>	Wheel Tracking Analysis
<i>XRD</i>	X-Ray Diffraction
<i>ZSV</i>	Zero Shear Viscosity