



STIFFNESS MODULUS PREDICTION MODEL FOR NATURAL NANO-ZEOLITE MODIFIED HRS-WC MIXTURES

*Eva Wahyu Indriyati¹, Bambang Sugeng Subagio² and Sony Sulaksono Wibowo³

¹Faculty of Civil and Environmental Engineering, Institut Teknologi Bandung, Indonesia; ¹Faculty of Engineering, Universitas Jenderal Soedirman, Indonesia; ^{2,3}Faculty of Civil and Environmental Engineering, Institut Teknologi Bandung, Indonesia

*Corresponding Author, Received: 23 March 2026, Revised: 15 May 2026, Accepted: 26 May 2026

ABSTRACT: The stiffness modulus is a critical design parameters in mechanistic methods for the structural evaluation of asphalt pavements. This study aims to develop a robust prediction model for the stiffness modulus (S_{mix}) of Hot Rolled Sheet-Wearing Course (HRS-WC) mixtures modified with natural nano-zeolite. Experimental tests were conducted using natural nano-zeolite contents ranging from 0.1% to 0.3%. The mechanical response was evaluated through Dynamic Shear Rheometer (DSR) tests for the binder and Indirect Tensile Strength (ITS) methods using the UMATTA tool for the mixtures at various temperatures. Results indicate that natural nano-zeolite modification significantly enhances the stiffness modulus of the binder compared to conventional 60/70 penetration grade asphalt, particularly by improving its elastic behavior. Statistical analysis led to the development of a predictive model: $S_{mix} = 2162.43 + 33.31 \text{ Sbit} + 727.16 \text{ NZ} - 23.26 \text{ VFA}$. The model identifies the asphalt stiffness modulus (S_{bit}), natural nano-zeolite content (NZ), and voids filled with asphalt (VFA) as primary independent variables. Validation results show that the predicted S_{mix} values align closely with laboratory data, with a ratio range of 0.630 to 1.395. This study concludes that the incorporation of natural nano-zeolite at low dosages (0.1%-0.3%) produces measurable improvements in mixture stiffness modulus at WMA production temperatures. The reinforcement mechanism is interpreted as being consistent with nano-filler behavior reported in prior literature, and warrants further microstructural verification.

Keywords: *Stiffness modulus of asphalt, Asphalt modification, Stiffness modulus of mixture, Natural nano-zeolite, Model*

1. INTRODUCTION

The performance and service life of asphalt pavements are fundamentally governed by the mechanical properties of asphalt mixture used in their construction. In recent years, increasing traffic volume, heavier axle loads, and fluctuations in ambient temperature have escalated the challenges faced by road infrastructure, leading to premature pavement distresses such as rutting and fatigue cracking [1].

The increasing demand for high-performance road infrastructure, driven by increasing traffic volumes and heavier axle loads, is challenging flexible pavement systems [2]. As the primary binder in asphalt mixtures, bitumen plays a critical role in determining the viscoelastic response and overall durability of the pavement [3]. However, conventional asphalt binders often struggle to resist permanent deformation at high temperatures and fatigue cracking at lower temperatures [4]. This issue has led to a paradigm shift in pavement engineering

toward the development of modified asphalt binders that can improve the mechanical properties and extend the service life of asphalt pavements [5].

In recent years, the integration of nanomaterials into asphalt technology has emerged as a transformative approach to address these structural limitations. Nanotechnology allows for the modification of bitumen at the molecular level, significantly improving its rheological properties [6-7]. Although synthetic polymers and nanomaterials have shown potential, high production costs and environmental impacts remain significant barriers [8]. Consequently, there is growing interest in sustainable mineral modifiers derived from natural sources, such as natural zeolites.

Zeolites are crystalline aluminosilicates characterized by a unique three-dimensional porous structure [9-10]. When processed into nano-sized particles, natural zeolite exhibits an exceptionally high surface area and cation exchange capacity, which facilitates better dispersion and stronger interfacial bonding within the asphalt matrix [11-12].

Previous studies have indicated that nano-zeolite can improve several aspects of asphalt mixture performance, including cracking and permanent deformation resistances [13-15]. However, the influence of this modifier on the stiffness modulus—a fundamental parameter for mechanistic-empirical pavement design—remains complex and not fully quantified.

The efficacy of nano materials in asphalt modification is primarily attributed to their high surface-area-to-volume ratio, which promotes the formation of a more stable bituminous mastic. Research has shown that the addition of nano-modifier can significantly alter the transition between the viscous and elastic states of the binder, thereby enhancing the complex shear modulus (G^*) across a wide range of service temperatures [16]. Unlike conventional modifiers, natural nano-zeolites have been reported in prior literature to exhibit active reinforcement characteristics attributed to their nano-scale particle size and porous framework structure, though direct microstructural verification remains an active area of research. Their porous framework allows for the partial absorption of light molecular components of bitumen, specifically the maltenes, which effectively increases the relative concentration of asphaltenes and leads to a more “structured” binder [12]. This phenomenon is critical for understanding the stiffening effect observed in modified mixtures.

The stiffness modulus is an important indicator of an asphalt mixture's ability to distribute loads and resist deformation [17]. While empirical testing provides local data, nano-modified mixtures require a more robust analytical approach. Predictive modelling approaches have been widely applied in asphalt pavement research, including fatigue resistance prediction [18] and permanent deformation characterization of modified mixtures [19]. Current predictive models often fail to account for the unique interaction between the pore structure of nano-zeolites and the chemical fractions of bitumen (saturates, aromatics, resins, and asphaltenes). This study aims to develop a comprehensive modelling framework. By synthesizing experimental data, this research provides deeper insights into the behavior of natural nano zeolite-modified mixtures, offering a path toward more resilient and sustainable pavement materials.

The present study makes three distinct contributions to the existing body of knowledge. First, it provides the first experimental dataset for natural nano-zeolite sourced from the Gunungkidul deposit in Central Java, Indonesia — a locally available natural mineral that has not previously

been investigated as a pavement modifier — incorporated at low dosages of 0.1%–0.3% by weight of binder in HRS-WC gap-graded mixtures. This dosage range is substantially lower than those reported for synthetic zeolite (1%–9%) [12] and other nano-modifiers (2.5%–10%) [16], reflecting the higher surface reactivity of nano-sized natural zeolite particles. Second, the study develops and validates a predictive model for mixture stiffness modulus ($Smix$) calibrated against UMATTA indirect tensile stiffness data — extending the modelling approach demonstrated for slag and RAP mixtures by Bethary et al. (2020) to nano-zeolite modified WMA systems. Third, the study identifies a threshold nano-zeolite content (0.1%) beyond which binder stiffness declines in a pattern consistent with nano-particle agglomeration behavior, providing the first quantitative evidence of this dosage threshold for natural zeolite in HRS-WC mixtures. The comparative of closely related studies is summarized in Table 1.

Table 1. Comparative summary of closely related studies

Study	Modifier type	Dosage range	Key output	Predictive model
Yao et al. (2013)	Nanoclay + carbon microfiber	2%-6% by binder weight	Rheological + chemical properties	None
Liu et al. (2023)	Synthetic zeolite	1%-9% by binder weight	Rheological properties	None
Yousif & Abed (2025)	Nano-silica, nano-chitosan, nano-clay	2,5%-10% by binder weight	Physical + rheological properties	None
Ahmadzadegan (2021)	Synthetic zeolite	0.1%-0.3%	Mechanical properties	None
Bethary et al. (2020)	Slag + RAP	Various	Resilient modulus	$Smix$ model
This study	Natural nano-zeolite	0.1%-0.3% by binder weight	Binder rheology + $Smix$	$Smix$ model

2. RESEARCH SIGNIFICANCE

This study addresses a gap in the characterization and modelling of natural nano-zeolite modified asphalt mixtures for tropical pavement applications. By providing the first experimental dataset for locally sourced Gunungkidul zeolite as a nano-scale binder modifier in HRS-WC mixtures, and developing a validated predictive model for mixture stiffness modulus calibrated against UMATTA stiffness data, this research supports more efficient mechanistic-empirical pavement design for warm mix asphalt

systems. The findings offer practitioners a practical modelling tool derived from standard mix design parameters, with broader implications for sustainable utilization of local mineral resources in road construction.

3. MATERIALS AND METHODS

3.1 Materials

The aggregate utilized in this study is crushed stone sourced from PT Sambas Wijaya, located in Purbalingga, Central Java Province. The specific asphalt mixture employed is a Hot Rolled Sheet Wearing Course (HRS-WC). All design criteria, including the aggregate gradation limits, aggregate properties, asphalt properties, and the asphalt mixture's Marshall Properties, are determined by strictly adhering to the Indonesian specifications [20]. Specifically, the final aggregate gradation was established by adopting the average values of the specified gradation limits, as illustrated in Figure 1.

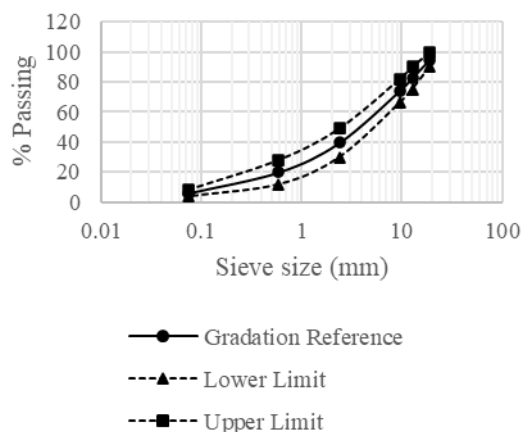


Fig.1 Aggregate gradation

The natural nano-zeolite was sourced from the Gunungkidul deposit in Central Java, Indonesia, and subjected to alkaline activation prior to incorporation into the asphalt binder. The activation procedure consisted of immersing the zeolite in 1 M NaOH solution and mixing under continuous stirring at 70°C for 3 hours. Following activation, the zeolite was washed twice with demineralized water, filtered through filter paper to remove residual, and dried at ambient temperature until constant mass was achieved. This activation procedure is intended to enhance the zeolite's surface reactivity by increasing the density of active exchange sites and modifying the surface charge characteristics, thereby promoting stronger interaction with the polar fractions of the asphalt binder.

XRF analysis of the activated zeolite identified the dominant chemical compounds as SiO₂ (76%), Al₂O₃

(15.8%), CaO (3.09%), Fe₂O₃ (2.91%), and K₂O (1.52%), yielding a SiO₂/Al₂O₃ molar ratio of approximately 4.8, tentatively consistent with clinoptilolite-type natural zeolite — the most commonly reported mineral phase in Indonesian zeolite deposits. Particle size was characterized through Dynamic Light Scattering using HORIBA SZ-100 on three replicate specimens at 25°C, yielding individual mean particle sizes of 322.9 nm, 326.4 nm, and 299.9 nm, with an overall mean of 316.4 nm. The particle size distribution is bimodal across all replicates, comprising a finer population centered at 123–184 nm and a coarser population at 362–511 nm, indicating inherent particle size heterogeneity that may predispose the material to agglomeration susceptibility at higher dosage levels. XRD mineral phase identification and BET surface area measurement were not performed in this study and are recommended as priorities for future material characterization.

The investigation involved two types of asphalt: original asphalt graded at 60/70 penetration supplied by Pertamina and natural nano-zeolite modified asphalt. The modification of asphalt binders with various additives has been shown to significantly alter mixture properties and performance characteristics [21]. The modified asphalt was prepared by incorporating natural nano-zeolite into the 60/70 penetration grade asphalt at varying proportions of 0.1%, 0.2%, and 0.3% by weight. These dosage levels were intentionally kept within a low range, as nano-sized particles exhibit significantly higher surface reactivity compared to conventional micro-sized modifiers, which typically require dosages of 0.25%-0.75% by mixture weight to achieve effective reinforcement [19, 21]. Preliminary observations and literature on nano-modifiers suggest that agglomeration becomes a limiting factor at higher dosages, making low-range incremental testing more appropriate for nano-zeolite binder modification.

The activated nano-zeolite was incorporated into the Pen 60/70 asphalt binder preheated to approximately 150°C using a conventional laboratory mechanical stirrer under continuous stirring for 30 minutes. No ultrasonic or high-shear pre-treatment was applied prior to mixing. The alkaline activation of the zeolite (1 M NaOH, 70°C, 3 hours) served as a surface pre-treatment to enhance dispersibility by increasing surface reactivity prior to binder incorporation. Homogeneity of the modified binder was assessed through visual inspection during the mixing and confirmed through the consistency of DSR complex shear modulus (G^*) measurements across replicate specimens at each NZ dosage level and aging condition. Specific mixer model, rpm, and

blade geometry were not recorded and are acknowledged as limitations of the protocol documentation. FTIR spectroscopy performed on modified binder specimens at all aging conditions and NZ dosage levels confirmed the presence of zeolite-associated spectral features — particularly in the Si-O-Si stretching region (approximately 1020–1031 cm^{-1}) and Al-O framework vibration region (approximately 808–864 cm^{-1}) — and showed progressive changes in binder chemistry with increasing NZ content, consistent with zeolite-binder chemical interaction.

The physical properties of the unmodified asphalt and the natural nano-zeolite modified asphalt was evaluated using established test methods. The full range of tests conducted were: penetration (SNI 2456:2011), softening point (SNI 2434:2011), specific gravity (SNI 2441:2011), flash point (SNI 2433:2011), viscosity (SNI 06-6441-200), and ductility (SNI 2432:2011). The resulting data set is summarized in Table 2.

Table 2. Properties of Pertamina asphalt graded at 60/70 penetration and natural nano zeolite modified asphalt

Test type	Asphalt Pen Grade 60/70	AsMod NZ0.1%	AsMod NZ0.2%	AsMod NZ0.3%
Penetration (dmm)	66.40	65.60	64.60	61.00
Softening point (°C)	57.50	58.00	57.50	57.50
Specific gravity	1.050	1.136	1.093	1.051
Flash point (°C)	350	349	338	341
Dynamic viscosity (Pa.s)	166.94	135.92	133.67	125.30
Ductility (cm)	> 100	> 100	> 100	> 100

The impact of using natural nano zeolite as a modifier on asphalt's rheological properties was assessed using the Dynamic Shear Rheometer (DSR), which provides the dynamic shear modulus (G^*) and phase angle (δ). DSR frequency sweep tests were performed on binder specimens under three aging conditions: (1) original unaged binder, (2) binder conditioned through Rolling Thin Film Oven Test (RTFOT) to simulate short-term aging during mixing and construction, and (3) binder conditioned through Pressure Aging Vessel (PAV) to simulate long-term in-service aging. DSR tests were performed on two replicate specimens ($n = 2$) for each binder variation and aging condition. The results reported represent the average of the two

replicates. While formal standard deviation calculation is not feasible from $n = 2$ replicates, the quality of time-temperature superposition achieved in the master curve construction – producing smooth, continuous curves without data scatter across all binder variations – provides implicit evidence of acceptable measurement repeatability. The complex shear modulus G^* obtained under each aging condition was used to calculate the corresponding asphalt stiffness modulus S_{bit} , ensuring that aging-induced changes in binder rheology are systematically represented within the model input parameters.

An analysis of the master curve (Figure 2), which depicts the material's initial sensitivity to temperature and loading frequency, revealed a substantial increase in the G^* value across the entire frequency range when natural nano zeolite was added. This suggests that the natural nano zeolite additive successfully reduces the asphalt's susceptibility to changes in loading frequency and temperature.

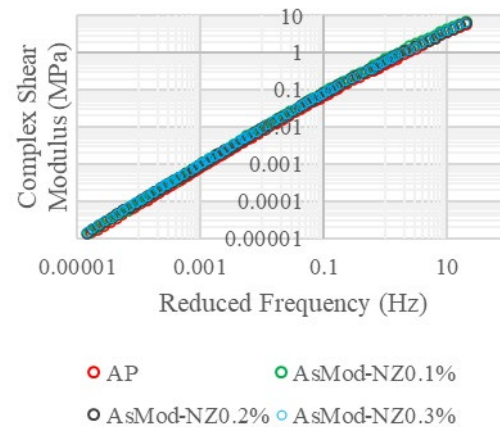


Fig.2 Master curve

As depicted in Figure 3, the stiffness modulus of the asphalt (S_{bit}) is related to its phase angle (δ). Under original conditions, the δ ranged from 68 to 90. Notably, the introduction of natural nano-zeolite reduced the δ value compared to the reference 60/70 penetration grade asphalt. A smaller phase angle indicates a shift toward more elastic behavior, which is consistent with this decrease. Moreover, the natural nano-zeolite modification resulted in a higher S_{bit} , translating to a stiffer asphalt material.

3.2 Methods

This investigation utilized four distinct mixture formulations, classified according to their specific binder composition. The optimum asphalt content for each mixture was determined using the Marshall method, and this value was subsequently adopted

for the resilient modulus testing of the mixtures. Mixing and compaction temperatures for each mixture were determined individually based on the viscosity-temperature relationship of the corresponding binder.

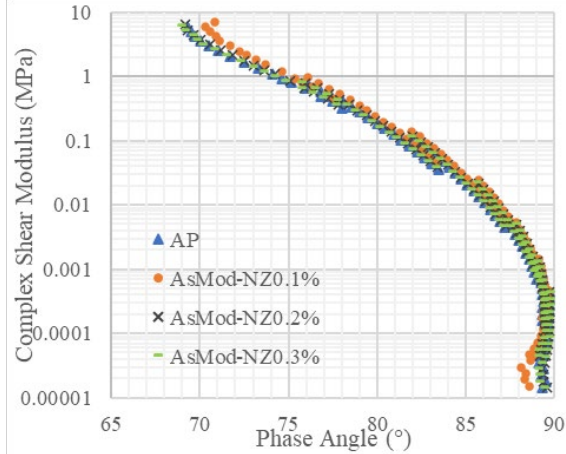


Fig.3 Black diagram

For WMA mixtures, the mixing and compaction temperatures were set at approximately 15% below those of conventional HMA. As the addition of nano-zeolite increases binder viscosity, the mixing and compaction temperatures of WMA mixtures increase progressively with NZ content, from 130°C and 120°C at NZ = 0.1% to 135°C and 123°C at NZ = 0.3%, respectively, as summarized in Table 3.

Table 3. The mixture variations

Mixture type	Natural nano zeolite content (%)	Mix. Temp (°C)	Comp. Temp (°C)	Optimum Asphalt Content (%)
HMA-AP	0	156	143	8
WMA-AsMod NZ0.1%	0.1	130	120	7.25
WMA-AsMod NZ0.2%	0.2	131	120	7.50
WMA-AsMod NZ0.3%	0.3	135	123	7.50

The mechanical characterization of asphalt mixtures through laboratory compaction has been established as a reliable approach for evaluating mixture performance [23]. The asphalt stiffness modulus was characterized through laboratory measurements conducted with a Dynamic Shear Rheometer (DSR) [24]. The complex shear modulus G^* obtained under each aging condition was used to calculate the asphalt stiffness modulus S_{bit} .

$$S_{bit} = 2(1 + \mu)G^* \quad (1)$$

Where S_{bit} is the stiffness modulus of asphalt in MPa, G^* is the complex shear modulus from the DSR test results in MPa, and μ is Poisson's Ratio. In accordance with established practice in asphalt binder rheology, a Poisson's ratio of $\mu = 0.5$ was assumed for all binder types in this study, reflecting the near-incompressible behavior of bituminous materials at DSR testing temperatures.

For comparison, the asphalt stiffness modulus is also determined theoretically, namely using the van der Poel nomogram as in Figure 4 and the Ullidtz equation listed in equation (2).

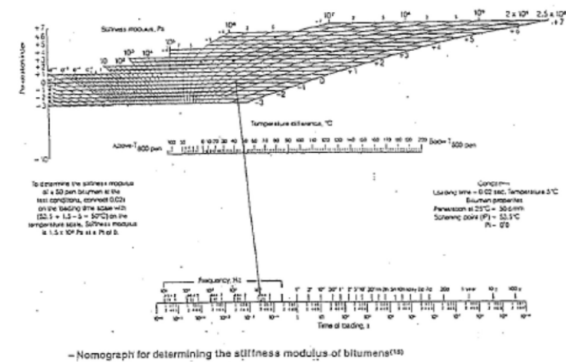


Fig. 4 Van der Poel Nomogram

$$S_{bit} = 1.157 \times 10^{-7} \times t^{-0.368} \times 2.718^{-PIr} \times (SP_R - T)^5 \quad (2)$$

Where

- S_{bit} = stiffness modulus of asphalt (MPa)
- t = loading time (second)
- PIr = recovered penetration index of asphalt
- SP_R = recovered softening point of asphalt
- T = temperature (°C)

Resilient modulus (S_{mix}) testing was performed using a Pavetest DTS-30 Universal Test Module following ASTM D4123-82, with a haversine loading pulse of 250 ms duration and a pulse repetition period of 3000 ms. Cylindrical specimens of 4-inch diameter were compacted using a Gyrotory Compactor with density targets derived from Marshall mix design results for each mixture variations. Testing was conducted at three temperatures: 25 °C, 35 °C, and 45 °C, with specimens conditioned at each target temperature prior to testing to ensure thermal equilibrium. Three replicate specimens ($n = 3$) were tested for each mixture variation and temperature combination, and all reported S_{mix} values represent the mean of three replicates.

Alternatively, the mixture's stiffness modulus can be predicted via various mathematical models. For comparative analysis within this study, the

Nottingham model was employed, which is defined by Eq. (3) and (4).

$$S_{mix} = S_{bit} \left\{ 1 + \frac{257.5 - 2.5VMA}{n(VMA - 3)} \right\}^n \quad (3)$$

$$n = 0.83 \log \left(\frac{4 \times 10^4}{S_{bit}} \right) \quad (4)$$

Where

S_{mix} = stiffness modulus of mixture (MPa)

S_{bit} = stiffness modulus of asphalt (MPa)

VMA = void in mineral aggregate (%)

4. RESULT AND DISCUSSION

4.1 Stiffness Modulus of Asphalt

The asphalt stiffness modulus (S_{bit}) was computed from the DSR complex shear modulus (G^*) using Eq. (1), with G^* values obtained at a reference frequency of 10 Hz, corresponding to a loading time of 0.016 seconds, representative of vehicle speed of 60-80 km/h. A Poisson's ratio of $\mu = 0.5$ was assume in accordance with established practice in asphalt binder rheology. Sensitivity analysis confirmed that variations of μ within a realistic range of 0.35-0.50 produces a maximum deviation in S_{bit} of 10.0%. The master curve presented in Fig. 2 presenting all four binder variations exhibit essentially identical master curve shapes, confirming that the frequency sensitivity of S_{bit} is consistent across NZ dosage levels.

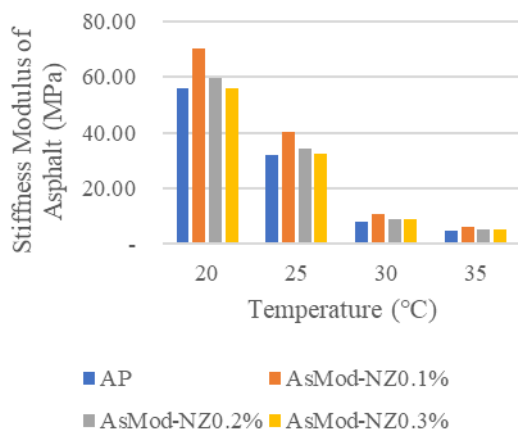


Fig.5 Stiffness Modulus of Asphalt

The influence of temperature and natural nano-zeolite (NZ) concentration on the Stiffness Modulus (S_{bit}) of 60/70 penetration asphalt is illustrated in Figure 5. The data reveal a consistent decay in S_{bit} as the temperature increases from 20 °C to 35 °C across

all samples. This phenomenon is attributed to the thermal activation of bitumen molecules, where increased temperature transitions the binder from a semi-solid elastic state to a more viscous fluid state. Notably, the modified binders (AsMod-NZ) maintained higher stiffness values compared to the base 60/70 penetration grade asphalt (AP) at elevated temperatures, suggesting improved rutting resistance in high-temperature climates. This is primarily due to the numerous pores and channels within zeolite [22]. These pores facilitate the adsorption of some of the oil fractions in the asphalt, ultimately contributing to increased hardness and stiffness modulus.

The incorporation of natural nano-zeolite at the tested dosage range produced a measurable increase in mixture stiffness modulus, with peak improvement observed at NZ = 0.1%, consistent with a reinforcing effect at low nano-particle concentrations. The highest reinforcement was observed at a 0.1% NZ concentration, which exhibited the superior S_{bit} across all experiment temperature. The observed decline in S_{bit} at NZ contents of 0.2% and 0.3% relative to the peak recorded at 0.1% is interpreted as being consistent with nano-particle agglomeration behavior reported in prior literature on nano-modified asphalt binders, wherein surplus nano-particles at higher concentrations tend to cluster, disrupting mastic homogeneity and reducing the reinforcing effect. It is acknowledged, however, that direct microstructural verification through SEM, TEM, or XRD analysis was not conducted in this study. Future research incorporating such characterization techniques is recommended to directly substantiate this mechanistic interpretation.

The S_{bit} improvement of 27.4%–30.6% observed at NZ = 0.1% is comparable in magnitude to the 30% dynamic modulus improvement reported by Yao et al. for Nanomer nanoclay at 2% dosage — a concentration 20 times higher than the optimal NZ dosage identified in this study. This dosage efficiency advantage is consistent with the higher surface reactivity of nano-zeolite relative to nanoclay at equivalent mass fractions, and aligns with the nano-scale particle size distribution characterized. The declining improvement at NZ = 0.2% (+5.7%) and NZ = 0.3% (+1.5%) mirrors the pattern reported by Yao et al. at higher nanoclay dosages, providing cross-study support for the agglomeration hypothesis. At the mixture level, the 10.8% improvement in S_{mix} at 25°C and 21.3% at 35°C for WMA-AMZeo0.1% relative to HMA-AP is consistent with the optimal performance at 0.1% NZ reported by Ahmadzadegan for WMA-SMA mixtures — despite differences in mixture type and performance metric — suggesting

that 0.1% represents a robust optimal dosage boundary for natural nano-zeolite across different HMA/WMA mixture systems. The substantially lower effective dosage compared to synthetic zeolite studies (1%–9%, Liu et al.) is attributed to the nano-scale particle size distribution of the Gunungkidul zeolite, which provides enhanced surface reactivity per unit mass.

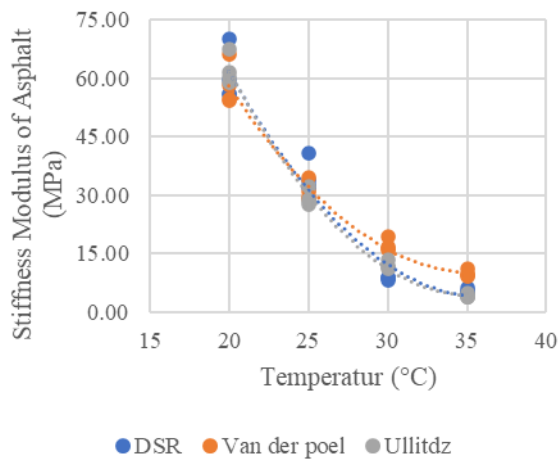


Fig.6 Theoretical and DSR Test Results of Asphalt Stiffness Modulus

There is a difference between the theoretically calculated Sbit value and the Sbit value obtained from laboratory testing using the DSR tool. The Sbit values from the Van der Poel nomogram and the Ullidtz equation are smaller than the DSR Sbit values. To bring the Van der Poel Sbit results closer to the DSR Sbit, an adjustment factor of 3.43 is required. Meanwhile, the Ullidtz Sbit requires an adjustment factor of 1.78.

Based on the two theoretical approaches used to determine the Sbit value, the Ullidtz equation is preferred for predicting the Sbit value of nano-zeolite-modified asphalt. This is because the influence of changes in penetration and softening point values due to the addition of nano-zeolite is more clearly illustrated using the Ullidtz equation.

4.2 Stiffness Modulus of Asphalt Mixture

The stiffness modulus of asphalt mixtures is affected by temperature. Based on Figure 7, the stiffness modulus values of mixtures using 60/70 penetration grade asphalt and natural nano-zeolite modified asphalt decreased as temperature increased. This is due to changes in the viscoelastic mechanical properties (from elastic to viscous) of the asphalt, which leads to a decrease in the stiffness modulus of the asphalt mixture [25].

In general, the incorporation of natural nano-

zeolite (NZ) into the bitumen significantly enhances the resilient modulus of the asphalt mixture compared to the conventional 60/70 penetration grade binder. As illustrated in Figure 7, which presents the dynamic response of mixture stiffness (S_{mix}) across a temperature spectrum of 25 °C to 45 °C, the reinforcement effect is particularly pronounced at lower temperatures. This initial enhancement suggests that NZ acts as a high-modulus modifier that strengthens the bitumen, thereby improving the overall load-bearing capacity of the asphalt pavement.

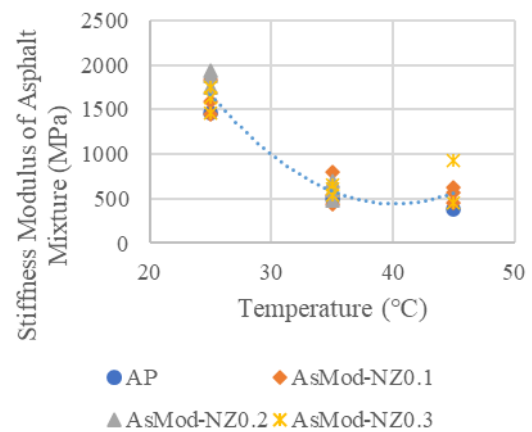


Fig.7 Stiffness Modulus of Asphalt Mixture

Interestingly, a divergence in performance was observed between the binder and the mixture phases at elevated temperatures. While the binder stiffness peaked at 0.1% NZ, the asphalt mixture exhibited superior retention at higher concentrations of 0.2% and 0.3% when subjected to thermal stress. Specially, at 45 °C, the AsMod-NZ 0.3% sample demonstrated a significantly higher resilient modulus than the control (AP). This phenomenon suggests that at higher concentrate, natural nano-zeolite effectively mitigates the effect of softened bitumen, reinforcing the aggregate structure and maintaining interlocks against permanent deformation.

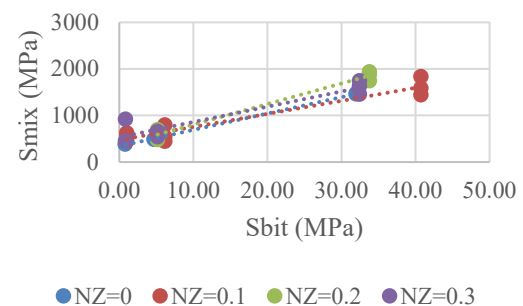


Fig.8 The relationship between S_{mix} and S_{bit} for each NZ dosage level

Fig. 8 presents the relationship between S_{mix} and S_{bit} for each NZ dosage level. The positive correlation between S_{mix} and S_{bit} across all NZ levels confirms that binder stiffness is the primary determinant of mixture stiffness in the tested conditions. At equivalent S_{bit} values, WMA-AMZeo0.1 mixtures yield consistently higher S_{mix} than WMA-AMZeo0.2 and WMA-AMZeo0.3, indicating that NZ contributes to mixture stiffness through mechanisms beyond binder stiffening alone — interpreted as a filler effect within the mastic phase that partially compensates for the reduced binder stiffness observed at higher NZ dosages. The volumetric properties of the mixture variations, summarized in Table X, show that VFA varies across mixture types in a manner consistent with the trend in S_{mix} , supporting the physical basis for VFA as a model input variable.

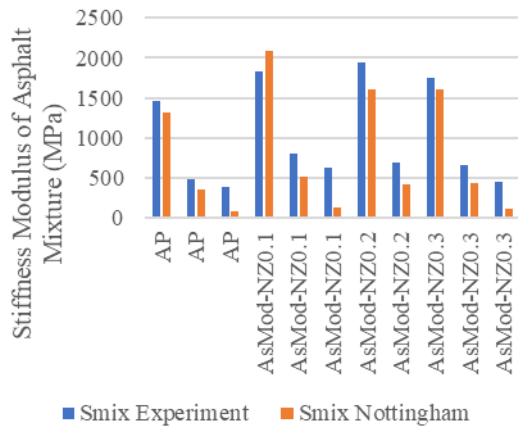


Fig.9 Comparison of Stiffness Modulus of Asphalt Mixture Experiment and Nottingham Model

Figure 9 shows that the theoretical stiffness modulus predicted by the Nottingham model, based on Eq. (2), is lower than that obtained from this experiment due to the different approaches used. The UMATTA test is based on the maximum applied load and resulting strain, while the Nottingham model predicts the stiffness modulus based on the asphalt stiffness modulus (S_{bit}) and the voids in mineral aggregate (VMA).

4.3 Prediction Model for Mixture Stiffness Modulus

The stiffness modulus of asphalt mixtures is a fundamental design parameter in flexible pavement planning [26]. Predicting the stiffness modulus value of the mixture is expected to support the flexible pavement design process. The development of the predictive model for the stiffness modulus of asphalt mixtures (S_{mix}) using nano-zeolite modified asphalt

utilizes a statistical approach, based on the following assumptions and conditions:

1. The nanomaterial used, nano-zeolite, acts as an asphalt modifier added to the binder before mixing with the aggregate.
2. The asphalt stiffness modulus (S_{bit}) used is derived from Dynamic Shear Rheometer (DSR) testing for each respective type of asphalt.
3. The asphalt mixtures tested are those utilizing the Optimum Asphalt Content (OAC) obtained from Marshall testing.

To develop the prediction model, regression analysis was employed, utilizing the mixture's stiffness modulus as the dependent variable alongside several independent factors [27]. Several specimens tested at 45°C produced anomalously low S_{mix} values with recoverable deformation readings at or below the instrument detection limit (approximately 0.00 μm), indicating measurement invalidity rather than actual material behavior. These data points — specifically all three replicates of WMA-AMZeo0.2 at 45°C and one replicate of WMA-AMZeo0.3 at 45°C — were excluded from the regression dataset on the basis of measurement invalidity.

Table 4. Mean and standard deviation of the retained data points

Mixture	Temperature (°C)	n	Mean S_{mix} (MPa)	SD (MPa)	CV (MPa)
HMA-AP	25	1	1464	-	-
HMA-AP	35	1	490	-	-
HMA-AP	45	1	383	-	-
WMA-AMZeo0.1	25	3	1621.7	196.3	12.1
WMA-AMZeo0.1	35	3	594.3	180.1	30.3
WMA-AMZeo0.1	45	3	548.0	85.0	15.5
WMA-AMZeo0.2	25	3	1854.7	97.6	5.3
WMA-AMZeo0.2	35	3	590.0	107.1	18.2
WMA-AMZeo0.3	25	3	1609.7	144.1	8.9
WMA-AMZeo0.3	35	3	604.0	60.3	10.0
WMA-AMZeo0.3	45	2	687.0	331.3	48.2

Based on statistical analysis with classical assumption tests, a model was obtained that meets the requirements..

$$S_{mix} = 2162.43 + 33.31 S_{bit} + 727.16 NZ - 23.26 VFA \quad (5)$$

Where

Smix = stiffness modulus of mixture (MPa)

Sbit = stiffness modulus of asphalt (MPa)

NZ = natural nano-zeolite (%)

VFA = void filled with asphalt (%)

The model indicates that increasing the asphalt stiffness modulus (Sbit) and natural nano-zeolite content will increase the mixture stiffness modulus (Smix). Meanwhile, increasing the VFA value will actually decrease the mixture stiffness modulus (Smix). Although temperature and loading frequency are not included as explicit independent variables, their combined influence on binder behavior is captured implicitly through the asphalt stiffness modulus (Sbit), which was determined from DSR frequency sweep tests across varying temperatures and loading times. Similarly, the influence of aggregate gradation on mixture behavior is represented implicitly through the VFA parameter, which is governed by the Voids in Mineral Aggregate (VMA) and thus reflects the volumetric consequences of gradation characteristics. The model is applicable within the boundary conditions stated herein, and validation against other mixture types and gradations is recommended in future studies.

The model was developed based on Smix test results with a test temperature range of 25 °C – 45 °C, an Sbit value range of 0.86 - 40.73 MPa, a natural nano-zeolite content range of 0% - 0.3%, and a VFA value range of 76.687% - 79.692%. The model has the following limitations:

1. The Smix test method refers to the ASTM D-4123 standard.
2. Aggregate gradation is a gap gradation with a maximum aggregate size of 19.1 mm.
3. The asphalt content is the optimum asphalt content, between 7% - 8%.
4. The nano-zeolite content is limited to 0% - 0.3% by weight of binder; the behavior at higher dosages remains to be investigated in future studies.
5. The VFA value is between the range of 76% - 80%.

It is acknowledged that the predictive model was developed based on 26 data points with three independent variables. While classical assumption tests confirm the statistical validity of the model within its defined applicability boundaries, the limited dataset size and the absence of an independent validation set represent constraints on the model's generalizability. Future studies are recommended to validate and refine this model using larger datasets and cross-validation

procedures.

The environmental sustainability of natural nano-zeolite as an asphalt modifier cannot be fully assessed from the findings of this study alone. The nano-zeolite was produced as a commercially processed product, and data regarding the energy consumption and environmental burden associated with its upstream extraction and nano-scale processing was not available. A comprehensive Life Cycle Assessment encompassing the full production chain – from raw material mining and nano-processing through mixture production, pavement service life, and end-of-life management – is required to determine whether the energy savings associated with reduced WMA production temperatures are sufficient to offset the environmental costs of nano-zeolite production. Beyond upstream environmental considerations, the hydrophilic character of natural zeolite introduces further interpretive boundaries for this study. The porous framework of zeolite particles has a tendency to retain moisture, which may affect binder–filler adhesion quality and accelerate aging-related degradation of the modified binder over time. Prior literature suggests that mineral fillers with high moisture affinity can facilitate water-induced debonding at the aggregate–binder interface, thereby elevating the risk of stripping and compromising long-term mixture durability [28]. In the present study, the pre-mixing moisture content of the nano-zeolite was not measured, and no dedicated moisture susceptibility evaluation at the binder level was performed. Consequently, the extent to which moisture-related interactions may have contributed to — or confounded — the observed rheological trends, particularly in specimens subjected to long-term aging simulation through PAV conditioning, cannot be determined. The findings of this study should therefore be understood within these boundaries, and subsequent research is encouraged to address these gaps through Life Cycle Assessment, systematic moisture quantification of the zeolite prior to incorporation, binder–aggregate adhesion evaluation, and performance comparisons against conventional mineral fillers under both dry and wet conditioning protocols.

The development of the predictive model for the stiffness modulus (Smix) was validated through classical statistical assumption tests to ensure its reliability and unbiased nature. The model satisfies all fundamental requirements:

1. Normality: The P-Plot analysis indicates that the data points align closely with the diagonal line, confirming a normal distribution of residuals.
2. Multicollinearity: The model is free from

multicollinearity issues, as indicated by correlation values between independent variables below 0.90 and a Variance Inflation Factor (VIF) value less than 10.

3. Homoscedasticity: The Glejser test resulted in a significance level greater than α (0.05), indicating the absence of heteroscedasticity.

Individual coefficient significance tests (t-test) yielded p-values of 0.000 for Sbit, 0.056 for NZ, and 0.554 for VFA. While Sbit is highly significant, the marginal significance of NZ and the non-significance of VFA at the conventional $\alpha = 0.05$ threshold reflect the limited statistical power associated with the 26-observation dataset rather than the absence of physical relationships between these variables and mixture stiffness. The overall model is statistically significant (F-test, $p = 0.000$), and all classical regression assumptions are satisfied.

The model demonstrates an exceptionally high degree of fit, with a Coefficient of Determination (R^2) of 0.9103 and an Adjusted R^2 of 0.8980. This implies that approximately 91% of the variability in the mixture's stiffness modulus can be explained by the three independent variables: asphalt stiffness (Sbit), nano-zeolite content (NZ), and voids filled with asphalt (VFA).

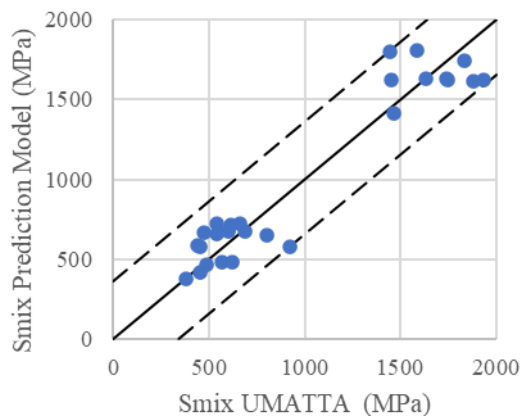


Fig.9 Model Validation

To validate the proposed model, laboratory test results for the mixture's stiffness modulus were compared against the model's predictions. As illustrated in Figure 9, the stiffness modulus values of the mixture calculated by the model are in close agreement with the laboratory test results. Validation of the predictive model yielded a ratio range of 0.630–1.395, with a mean ratio of 1.031 and a median of 0.978, indicating the absence of systematic bias across the dataset. The extreme ratio values were observed at boundary conditions of the experimental domain — specifically at the highest NZ content (0.3%) combined with the highest testing temperature (45°C), and at an intermediate NZ

content (0.2%) at 35°C — where non-linear viscoelastic interactions are most pronounced.

The predictive model proposed in this study — $Smix = 2162.43 + 33.31 Sbit + 727.16 NZ - 23.26 VFA$ — should be regarded as a preliminary model developed from a dataset of 26 observations. While the model satisfies classical regression assumptions and demonstrates high overall fit ($R^2 = 0.9103$, Adjusted $R^2 = 0.8980$), the individual significance of NZ ($p = 0.056$) and VFA ($p = 0.554$) coefficients is limited by the small dataset size and associated statistical power constraints. The model is recommended for use as a first-order estimation tool within the defined applicability boundaries, pending validation against a larger and independent dataset encompassing a wider range of mixture compositions, temperatures, and NZ dosage levels.

The applicability of the findings and the predictive model developed in this study is subject to the following boundary conditions: (1) the agglomeration mechanism proposed to explain the threshold effect in binder stiffness is interpreted based on consistency with existing literature, as direct microstructural evidence was not obtained; (2) the predictive model was developed for HRS-WC gap-graded mixtures with a maximum aggregate size of 19.1 mm, consistent with Indonesian national specifications, and its applicability to other mixture types, gradation curves, or aggregate sizes has not been verified. Future studies are recommended to address both of these limitations through microstructural characterization and multi-gradation experimental validation.

5. CONCLUSION

Based on the analysis and discussion regarding the stiffness modulus prediction model for asphalt mixture modified with natural nano-zeolite, the following conclusion are drawn:

1. Natural nano-zeolite modification significantly enhances the stiffness modulus of asphalt compared to standard 60/70 penetration grade. While both the Van der Poel nomogram and the Ullidtz equation yield lower stiffness values than direct DSR testing, the Ullidtz equation has been proven to be the more accurate predictive model for this modified binder.
2. The stiffness modulus of the mixtures exhibited an inverse relationship with temperature, resulting from the asphalt's transition from elastic to viscous behavior. The reinforcing effect of natural nano-zeolite was most pronounced at lower temperatures; however, at elevated temperatures, the improvement in resilient modulus was most notable at the 0.1%

modification level.

3. The predictive model proposed in this study — $Smix = 2162.43 + 33.31 Sbit + 727.16 NZ - 23.26 VFA$ — should be regarded as a preliminary model developed from a dataset of 26 observations. While the model satisfies classical regression assumptions and demonstrates high overall fit ($R^2 = 0.9103$, Adjusted $R^2 = 0.8980$), the individual significance of NZ ($p = 0.056$) and VFA ($p = 0.554$) coefficients is limited by the small dataset size and associated statistical power constraints. The model is recommended for use as a first-order estimation tool within the defined applicability boundaries, pending validation against a larger and independent dataset encompassing a wider range of mixture compositions, temperatures, and NZ dosage levels.
4. The predictive model is applicable within the following explicitly defined boundary conditions, beyond which its predictions should not be extrapolated without further validation: (1) mixture type — HRS-WC gap-graded asphalt mixture conforming to Indonesian national specifications with a maximum aggregate size of 19.1 mm; (2) binder type — Pen 60-70 asphalt binder modified with NaOH-activated natural nano-zeolite from the Gunungkidul deposit or mineralogically similar sources; (3) nano-zeolite content — 0% to 0.3% by weight of binder; (4) volumetric properties — VFA within the range of approximately 76%-80% and VMA within the range of 18%-22%; (5) test temperature — 25°C to 45°C, with the caveat that model applicability at 45°C is limited to NZ = 0.1% and NZ = 0.3% conditions; (6) Sbit range — 0.5-41.0 MPa as determined from DSR frequency sweep at 10 Hz reference frequency. The model should be regarded as a preliminary predictive tool, and its application outside these ranges carries significant uncertainty.
5. Validation results demonstrated that the Smix values calculated from the model closely align with laboratory test results, yielding an average ratio in the range of 0.630-1.395. This indicates that the proposed model is a robust tool for supporting the flexible pavement design process involving nano-modified materials.

6. ACKNOWLEDGMENTS

The authors extend their heartfelt gratitude to the Ministry of Research and Technology, for the generous funding and support, which made this research possible and impactful.

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