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Research Article

Surface-Modified Nanoclays for Enhancing Resistance to Moisture Damage in Hot Mix Asphalt

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Abstract

Previous research indicated that nanomaterials have potential in improving pavement properties, particularly moisture resistance. This study evaluated the effectiveness of nanoclays in enhancing the resistance of Hot Mix Asphalt (HMA) to moisture damage and compared its performance to standard modifiers. Asphalt binder modified using four additives was tested using a Dynamic Shear Rheometer (DSR) before and after being aged in a Rolling Thin Film Oven (RTFO): two surface-modified nanoclays and two liquid anti-stripping chemicals (HP+ and LOF 6500). The DSR and RTFO tests showed that the two nanoclays had a stiffening effect on the binder, while both liquid antistripping agents had the opposite effect, decreasing both the elastic and complex modulus of the binder. After RTFO aging, similar trends were observed, except the binder had become much stiffer in all cases. HMA designed employing the Superpave mix design procedure was tested for moisture sensitivity in accordance with AASHTO T-283. The dry tensile strength for the two nanoclays and LOF 6500 modified mixes were higher than the control mix. However, all modified mixes resulted in wet tensile strengths that were higher than the control. The tensile strength ratios for all modified mixes were also higher than the control and exceeded the Superpave mix design method minimum of 0.80. Evaluation of these additives in the field would further benefit asphalt pavement research.

Keywords

Nanoclay, Moisture Resistance, Hot Mix Asphalt, Anti-Stripping

1. Introduction

Various rehabilitation and maintenance treatments are utilized by Departments of Transportation (DOTs) to treat asphalt pavements experiencing moisture-related damage. The intrusion of water is responsible for breaking the aggregate-binder bond in asphalt pavements [1]. Asphalt pavements that face water infiltration frequently lose aggregates. The water's chemical attraction to the aggregates weakens the bond between the asphalt binder and aggregates, leading to the washing away of the binder [2]. The weakening of the bond coupled with the repetitive traffic loading leads to progressive aggregates dislodgement. This failure can result in different forms of distress like rutting, shoving, raveling, or cracking [3]. A National Cooperative Highway Research Program (NCHRP) study conducted in 1991 reported that majority of state and provincial DOTs in North America, who responded to the survey, reported moisture-related damage in

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their pavement networks [4]. The NCHRP study reported that rutting, bleeding, and cracking were some of the premature distresses related to moisture susceptibility.

Moisture damage is primarily caused by the loss of adhesion [1, 5]. In the case of hydrophilic aggregates, water absorption causes the stripping of binder from the aggregate surface, leading to potholes and under-layer failure [5].

When pavement is exposed to moisture, it can lead to premature failure due to the process of stripping. This phenomenon usually starts at the lower layer of the HMA and gradually moves upward over time. However, it can be hard to identify stripping as it can also cause cracking, rutting, and corrugations. If stripping initiates from the surface and moves downwards over time, it is termed as raveling [6-8].

Exposure to moisture can result in a considerable decline pavement surface condition, which in- turn leads to an escalation in maintenance expenses. Inadequate drainage that permits water infiltration is among the primary reasons for moisture damage. Although subgrade drainage is essential, the conventional repair technique involves the removal and replacement of the pavement [6]. Nevertheless, these repair and maintenance procedures can be quite expensive. Therefore, researchers have explored the possibility of incorporating additives/modifiers to enhance pavement resistance to the stripping of the binder.

A wide range of chemical additives have been utilized to enhance the bond between the binder and aggregate. These chemicals are typically mixed with the binder, either before or during mix production [9]. Anti-stripping additives are known to improve the aging characteristics of the binder, enhance resistance to temperature susceptibility, and have a tendency to soften the binder [10]. Liquid antistripping agents aid in enhancing the moisture resistance of HMA by reducing the surface tension between the aggregate surface and the asphalt binder, thereby augmenting the binder's adhesion to the aggregate surface.

Solid additives such as hydrated lime, Portland cement, fly ash, flue dust, and polymers have also been utilized to enhance moisture resistance in hot-mix asphalt mixtures. Typically, these additives are incorporated into the aggregate before being blended with the binder during the HMA production process. Nonetheless, hydrated lime or Portland cement can also be added during the drum mixing operation, at the point of entry of the binder to the heated aggregate [11]. Hydrated lime can neutralize the acidity in the asphalt binder and enhance the bond between the binder and the aggregate. When the aggregate is treated with hydrated lime, both anionic and cationic surfactants, which are naturally present in the bitumen, can strongly adhere to calcium ions.

Adding 1.0% class F fly ash to the asphalt mix resulted in a resilient modulus comparable to that of the control mixture, but slightly lower than that of HMA treated with hydrated lime [12]. The tensile strength ratio (TSR) tests showed a 15% higher ratio in tensile strength over the control mixture, while hydrated lime increased the TSR by 25% over that of the control mix. Furthermore, By mixing asphalt binder with cement kiln dust (CKD) before introducing it to the aggregate, the requirements for asphalt binder can be significantly reduced. CKD also has the potential to replace hydrated lime and decrease moisture damage in pavements due to its high lime content, as confirmed in a study on various mineral fillers [12]. Addition of 1.0% CKD to the asphalt mix produced a TSR within a few percent of the hydrated lime variations and nearly 25% higher than the untreated control mixture.

Research studies have shown that nanomaterials have the potential to provide greater improvements in moisture resistance compared to commonly used materials. Specifically, modification of asphalt binders with nanoclays has been found to enhance the performance of the mix in various ways. For example, it can increase the dynamic shear complex modulus, reduce the strain failure rate, improve rutting resistance, reduce the penetration value, increase the softening point temperature and viscosity, and enhance the fatigue life of the asphalt mix [13-24]. Nanoclays can also improve the cohesion of the asphalt binder, thereby increasing the material's ability to heal micro-cracks [25]. Additionally, amine modified nanoclays contain alkyl amines, which are commonly used in antistripping liquids to reduce moisture sensitivity in HMA.

In 2016, Ameri et al. [26] investigated the effects of montmorillonite nanoclay, Cloisite 15A, and Cloisite 30B modified with organic chemicals, added at a dosage of 2-6% by weight of bitumen, on moisture resistance. Their findings revealed that both nanoclays improved the resistance to moisture damage, as the TSR for the modified samples was consistently higher than the control mixture with a TSR of 80%. Moreover, it was observed that Cloisite 30B at 6% showed the best results, with a TSC above 95%. The addition of polysiloxane-modified montmorillonite nanoclay at a dosage range of 0.5 to 1.8% by weight of bitumen was found to improve the stripping resistance of the asphalt mixtures when exposed to deicer solutions at varying concentration levels, regardless of the type of deicer solution used [27].

A comparative evaluation of the effectiveness of nanoclays in enhancing moisture damage resistance compared to commonly used stripping resistance additives, such as hydrated lime and liquid antistripping agents, is lacking in the literature. Additionally, other types of surface-modified nanoclays may offer superior performance compared to those already studied in the literature. The current study focused on amine-modified nanoclays because alkyl amines are widely used chemical additives to enhance moisture resistance in asphalt binders. The study aimed to achieve two specific objectives: (a) assess the effectiveness of novel types of surface-modified nanoclays as modifiers to reduce the moisture sensitivity of hot mix asphalt; and (b) compare the performance of the nanoclays used in this study with hydrated lime and liquid anti-stripping agents, which are commonly used by some DOTs to reduce the moisture sensitivity of hot mix asphalt.

2. Materials and Testing

2.1. Aggregates

The aggregate selected for the HMA design were subjected to several crucial property and performance tests. The tests conducted included evaluations of bulk and apparent specific gravities, as well as durability, angularity, and clay content tests. Table 1 provides the specific gravity and absorption properties of the aggregate. The selected aggregate had a Sand Equivalent (SE) value of 85% and a 24% loss in the Los Angeles abrasion test.

2.2. Additives

Surface-modified nanoclays, lime-slurry, and two amine-based liquid anti-stripping agents were tested as additives to enhance moisture resistance. The surface-modified nanoclays were procured from Sigma-Aldrich (Table 2). The aggregate treated with lime slurry (1.3%) was received ready for testing from the supplier, and the amine-based chemicals (HP+ and LOF 6500) were obtained from ArrMaz Custom Chemicals. All the additives, except for the lime-slurry treated aggregate, were added to the binder while heated and mixed thoroughly. The percentages at which the additives were added are provided in Table 3. Laboratory tests conducted on aggregate included specific gravities, durability, angularity, and clay content.

Table 1. *Specific Gravity of Coarse and Fine Aggregates.*

Aggregate Type	Bulk Specific Gravity	Bulk Specific Gravity (SSD)	Apparent Specific Gravity	Absorption, %
Coarse[1]	2.58	2.63	2.71	1.80
Fine[2]	2.40	2.54	2.79	5.00

[1]Retained on Sieve #4 and [2]Passing Sieve #4

Table 2. *Types and properties of nanoclays used in the study.*

Nanoclay	Properties
Nanclay01: Nanoclay, surface modified with trimethyl stearyl ammonium	Montmorillonite clay Contains 25-30 % by wt. trimethyl stearyl ammonium
Nanoclay02: Nanoclay, surface modified with octadecylamine and aminopropyltriethoxysilane	Montmorillonite clay Contains 13-35 % by wt. octadecylamine And 0.5 5 % by weight aminopropyltriethoxysilane

Table 3. *Additive concentrations[1].*

Nanoclay, %	Liquid Antistripping, %
1.0	0.25
2.0	0.50
4.0	0.75
6.0	---

[1] Percentage of binder weight

2.3. Asphalt Binder

In this study, Performance Grade 64-10 (PG 64-10) with a specific gravity of 1.02 was used. The virgin binder was evaluated using Dynamic Shear Rheometer (DSR) both before and after being aged in Rolling Thin Film Oven (RTFO). The asphalt binder was heated and blended with additives before undergoing DSR testing. DSR testing was carried out again after mixing the binder with antistripping additives at different percentages, and the outcomes are shown in Table 4.

Table 4. The DSR Test Results.

Additive Type	Additive content, %	Complex Modulus, kPa	Phase Angle, °	Elastic Modulus, kPa
Unaged Binder				
Control	0.0	1.43	87.1	0.076
Nanoclay01	1.00	1.82	86.5	0.111
	2.00	1.86	86.2	0.123
	4.00	1.90	86.1	0.129
	6.00	2.27	86.0	0.158
Nanoclay02	1.00	1.82	86.7	0.104
	2.00	1.85	86.4	0.115
	4.00	1.91	86.3	0.118
	6.00	2.32	86.2	0.153
HP Plus	0.25	0.449	85.6	0.034
	0.50	0.331	84.8	0.030
	0.75	0.207	82.5	0.027
LOF 6500	0.25	0.837	89.1	0.128
	0.50	0.467	88.8	0.101
	0.75	0.345	87.7	0.136
RTFO Aged Binder				
Control	0.0	3.08	84.5	0.276
Nanoclay01	1.00	3.70	83.9	0.393
	2.00	4.95	83.3	0.578
	4.00	7.46	84.0	0.779
	6.00	9.96	83.6	1.110
Nanoclay02	1.00	3.88	84.3	0.344
	2.00	5.20	84.0	0.543
	4.00	7.83	83.8	0.845
	6.00	10.46	83.6	1.166
HP Plus	0.25	2.90	85.4	0.228
	0.50	2.50	86.1	0.172
	0.75	1.75	87.1	0.090
LOF 6500	0.25	2.37	86.8	0.133
	0.50	2.24	87.0	0.126
	0.75	0.91	89.2	0.013

2.4. Hot Mix Asphalt

The SuperPave mix design procedure was used to design the HMA, which involved selecting the appropriate aggregate and determining the optimal binder content. Three different aggregate gradations (listed in Table 5) were considered, with the California Department of Transportation (CALTRANS) gradation requirements for 12.5-mm (0.50-inch) HMA adopted. The HMA process ultimately yielded an optimal binder content of 5.75% using the intermediate blend (#2) as the design blend.

In this study, a total of 90 specimens were tested for each mix, divided into two subsets of three specimens each, with a target air voids content of 7.0% ±0.5% at the optimum binder content. The first subset was tested in an unconditioned state, while the second subset was partially vacuum-saturated (with a degree of saturation of 70% to 80%) and subjected to one freeze-thaw cycle according to AASHTO T-283. Two different methods were used to calculate the TSR; first, by dividing the Indirect Tensile Strength (ITS) of conditioned specimens by the ITS of unconditioned specimens for each additive combination, which is the common practice, and second, by dividing the ITS for the modified mixes by the ITS of the unconditioned mix (control mix), in order to standardize the comparison. This second method, referred to as $TSR_{normalized}$, was explored in this study.

Table 5. *Sieve Analysis Results for Different Aggregate Blends.*

Sieve Opening, mm	**19**	**12.5**	**9.5**	**4.75**	**2.38**	**0.60**	**0.075**
Range, % Passing	100	90 - 98	70 - 90	42 - 58	29 - 43	10 - 23	2 – 7
Coarse Blend	98	85	62	53	42	24	4.0
Intermediate Blend	100	96	82	44	31	18	4.5
Fine Blend	100	97	89	69	56	32	6.6

3. Results and Discussion

3.1. Asphalt Binder

The results of Dynamic Shear Rheometer (DSR) tests conducted on the asphalt binder before and after Rolling Thin Film Oven (RTFO) aging are discussed. The key properties evaluated are the complex modulus, phase angle, and rutting factor. It is important to consider multiple performance indicators of the asphalt binder for various applications at different temperatures. The rutting factor, represented by G*/sinδ, indicates the binder's ability to recover deformation after load removal and its resistance to high-temperature deformation. A high rutting factor corresponds to better resistance to failure in rutting.

Figures 1 and 2 depict the rutting parameter (G*/sinδ) values of the binder under different aging conditions and additive types/contents. The data show that the addition of additives results in an increase in the rutting parameter (G*/sinδ), indicating enhanced resistance to rutting deformation. Figure 1 reveals that the rutting factor (G*/sinδ) was raised beyond the maximum limit of 0.249 psi specified by Superpave mix design due to the two nanoclay additives. However, G*/sinδ for nanoclays with concentrations ranging from 1% to 4% remains close to the maximum limit of 0.249 psi. Meanwhile, the two liquid antistripping resulted in a reduction in the rutting factor below the minimum limit specified by Superpave mix design. For the binder aged in RTFO, Figure 2 shows that the G*/sinδ exceeds the minimum requirement of 0.319 psi for virgin binder and binder modified with the two nanoclays at 1% to 6% concentrations. Nevertheless, G*/sinδ for binder modified with liquid antistripping's concentration of 0.25% and 0.5% was slightly above the minimum of 0.319 psi.

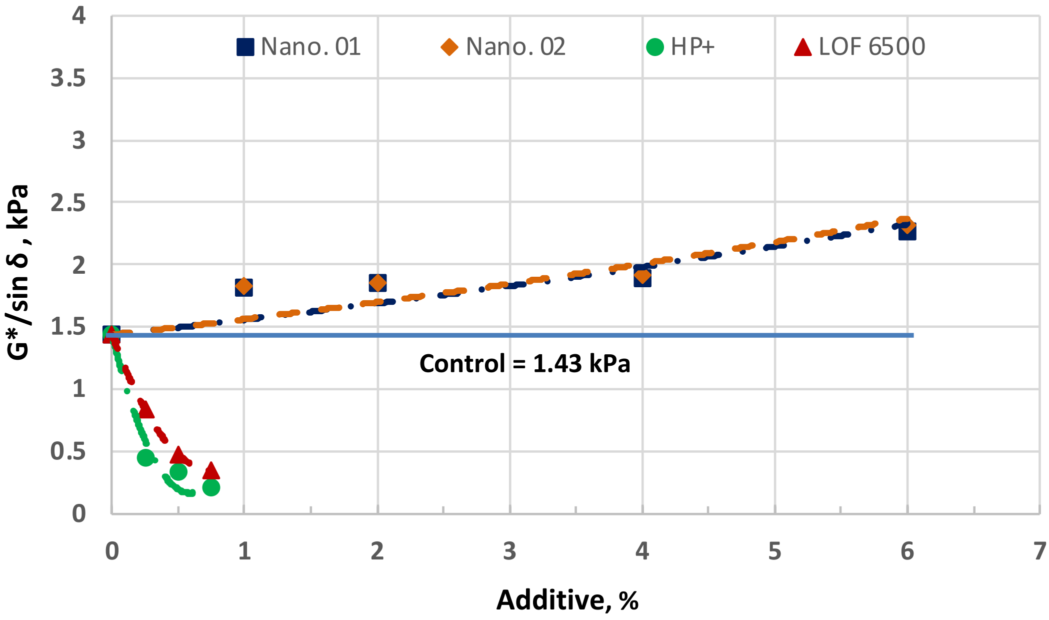

Figure 1. Rutting factor for virgin/unaged asphalt binder.

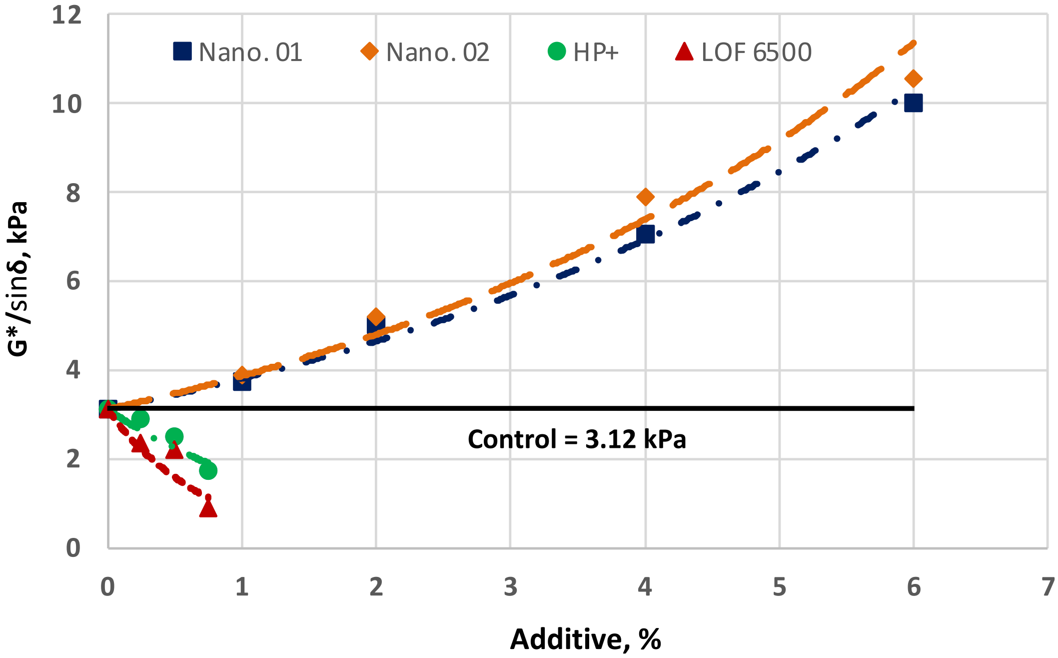

Figure 2. Rutting factor for RTFO-aged asphalt binder.

3.2. HMA Moisture Sensitivity

3.2.1. ITS for Unconditioned/Dry Mixes

Figure 3 presents the results for the ITS for the unconditioned specimens. The data indicates that the addition of nanoclay modifiers, specifically nanoclay01 and nanoclay02, resulted in higher tensile strength in HMAs. The ITS increased at a small rate as the percentage of nanoclay01 or nanoclay02 increased, with an optimum percentage of approximately 3% for both additives. In contrast, HMAs containing lime-treated aggregate showed ITS that is slightly lower than that for the control mix, with testing performed only at a lime content of 1.3%. The performance of HMA treated with liquid antistripping was mixed. HMA treated with LOF 6500 exhibited an increase in ITS, with an observed optimum at approximately 0.5%. In contrast, HMA treated with HP+ liquid antistripping had lower ITS than that for the control mix. It is worth noting that all mixes tested resulted in dry tensile strengths above the minimum of 100 psi specified by the 2018 California Department of Transportation (Caltrans) Standard Specifications.

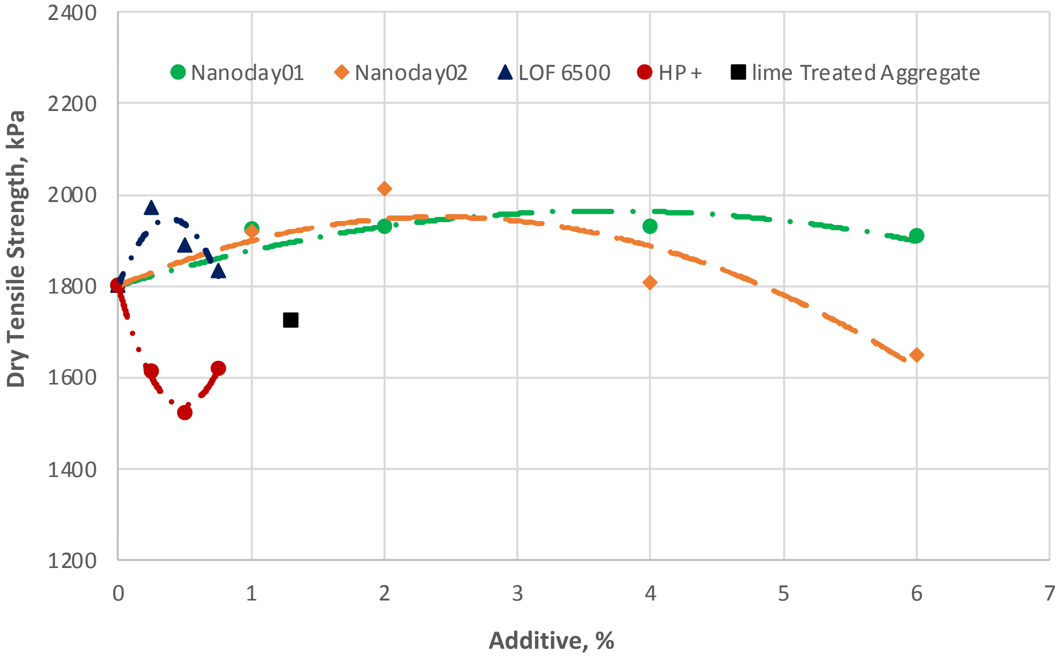

Figure 3. *ITS for Unconditioned/dry Specimens (Note: Mixes with 0.0% additives is the control mix with ITS = 1801 kPa).*

3.2.2. ITS for Conditioned Mixes

Figure 4 presents the comparison of ITS for conditioned specimens. As can be seen in Figure 4, all mixes tested showed similar performance trends. HMAs treated with nanoclay01 and nanoclay02 exhibited higher strengths than the control mix, except for the nanoclay02 mix with 6% nanoclay. It is worth noting that the optimum additive percentages for nanoclay01 and nanoclay02 were approximately 2.5% and 3.5%, respectively. In contrast, the HMA mix with lime-treated aggregate performed better than the control mix. Both liquid antistripping agents showed similar trends, with an observed optimum antistripping percentage of approximately 0.5%. It is worth noting that all mixes resulted in wet tensile strengths greater than the minimum of 70 psi specified by Caltrans.

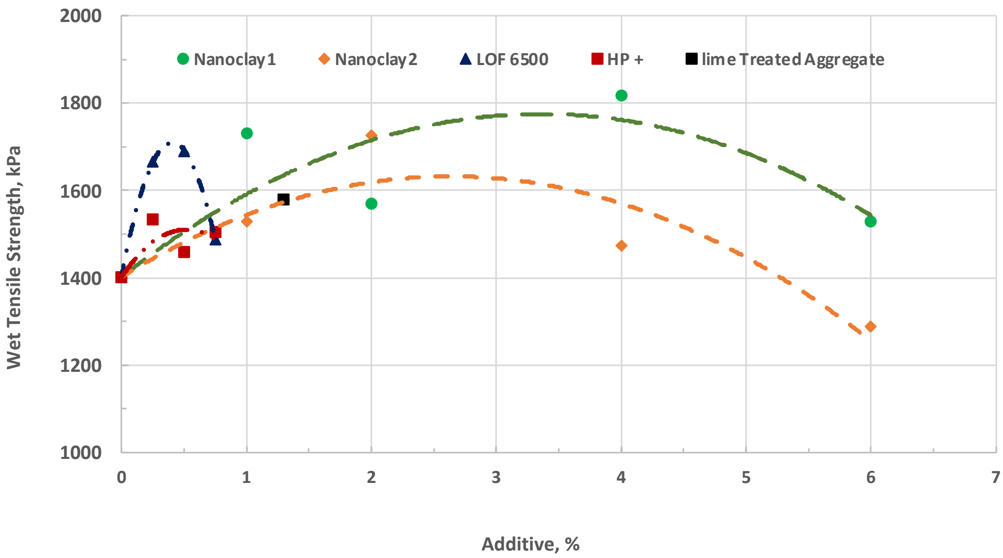

Figure 4. *ITS for Conditioned/Wet Specimens (Note: Mixes with 0.0% additives is the control mix with ITS = 1402 kPa).*

3.2.3. Tensile Strength Ratio (TSR)

The results of the TSR (ITS for conditioned specimens divided by ITS for unconditioned speciemns) for each subset of specimens are presented in Figure 5. Figure 5 shows that all modified mixes outperformed the control mix and exceeded the minimum requirement of 0.70 specified by Caltrans. However, the TSR for the control mix was slightly below the 0.80 specified by Superpave mix design. HP+ liquid antistripping was found to be the most effective modifier among all the modifiers investigated in this study. It should be noted that, from among the modified mixes tested in this laboratory study, HMA modified with Nanoclay02 resulted in the least improvement in TSR. For all additives tested (other than the hydrated lime) an optimum additive percentage can be observed in the figure. It should be noted that only one lime percent (1.3%) was tested in this study.

In order to standardize the TSR, the ratio of the ITS for the conditioned specimens to the ITS for the unconditioned control mix was calculated. This ratio is referred to as $TSR_{normalized}$ throughout the study and is presented in Figure 6. The results indicate that $TSR_{normalized}$ for all modified mixes outperformed the control mix, with all mixes exceeding the 0.80 minimum specified by Superpave (Figure 6). However, the $TSR_{normalized}$ and the optimum modifier content varied slightly for each mix. Table 6 presents the comparison between the TSR calculated using the two approaches.

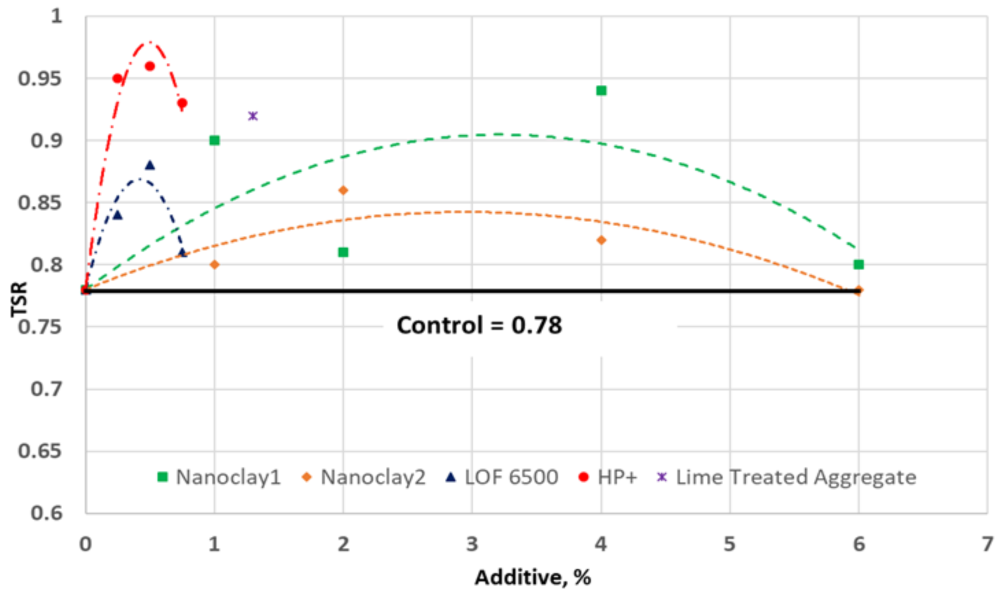

Figure 5. *TSR for Modified HMA.*

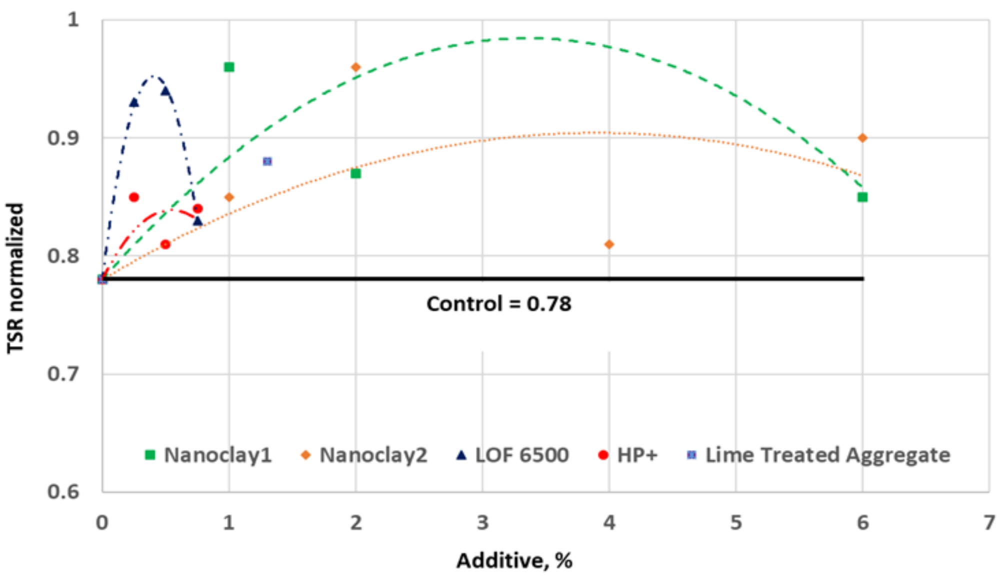

Figure 6. *$TSR_{normalized}$ for modified HMA.*

Table 6. *Comparison between the two approaches used in calculating the tensile strength Ratio.*

Modifier	TSR/Modifier Optimum Content, %	$TSR_{NORMALIZED}$/Modifier Optimum Content, %
Nanoclay01	0.90 / 2.5	0.97 / 3.5
Nanoclay02	0.84 / 3.0	0.87 / 3.5
Lime Slurry	0.92 / NA[1]	0.88 / NA
LOF 6500	0.87 / 0.5	0.95 / 0.4
HP+	0.96 / 0.5	0.83 / 0.5

[1] Not applicable since only one lime slurry percentage was used

The application of the two methods for calculating TSR showed mixed outcomes. $TSR_{normalized}$ produced higher results compared to TSR for three of the mixes (nanoclay01, nanoclay02, and LOF 6500) while lower results were obtained for the other two mixes (lime-treated and HP+). These findings highlight the need for discussion among the pavement researchers to reach a consensus on the appropriate approach for analyzing the outcomes of AASHTO T283.

4. Conclusions

This laboratory research investigated the effectiveness of various to enhance the resistance of HMA to moisture-related damage. Standardized testing procedures were employed to test the physical and chemical properties of aggregates. Also, rheological properties of asphalt binder were tested for both the virgin and modified binder. The DSR test results showed that the two nanoclay additives improved the stiffness of the asphalt binder. Higher concentrations of nanoclays led to even greater stiffness. However, liquid antistripping additives resulted in a significant reduction in the binder stiffness. With percentages higher than 0.5% resulted in a reduction in binder stiffness below the minimum requirement specified by Superpave. The additives tested in this study, except for HP+, resulted in dry tensile strengths that were higher than the control mix. Additionally, all additives (including HP+) resulted in higher wet tensile strength than the control mix. It is recommended to use TSR calculated as a ratio of the dry tensile strength of the control mix as an indicator of moisture resistance. The researchers believe that the use of $TSR_{normalized}$ approach introduced in this study, as the aim of this study is to enhance the control mix using different additives/modifiers, all comparisons should be made in reference to the mix targeted for improvement.

Abbreviations

AASHTO: American Association of State Highways and Transportation Officials
CALTRANS: California Department of Transportation
CKD: Cement Kiln Dust
DOT: Department of Transportation
DSR: Dynamic Shear Rheometer
HMA: Hot Mix Asphalt
ITS: Indirect Tensile Test
NCHRP: National Cooperative Highway Research Program
RTFO: Rolling Thin Film Oven
SSD: Saturated Surface Dry
TSR: Tensile Strength Ratio

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Conflicts of Interest

The authors declare no conflicts of interest.

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