Size Effect of Sub Nano-scaled Hydrated Lime on Selected Properties of **HMA**

Junan Shen¹⁺, Baoshan Huang², Xian Shu³, and Boming Tang⁴

Abstract: Hydrated lime (HL) has been used as an additive in hot mix asphalt (HMA) mainly as an anti-stripping agent and mineral filler. The properties of the HMA added with the HL improve through the reaction of the HL with asphalt binder. This study investigates the size effect of HL on selected properties of HMA. First, an HL in HMA plant of Georgia, USA, was used for the production of sub nano-sized particles using an LA Abrasion machine in the laboratory. The particle size of the produced HL was measured through a microscope. Second, selected properties of asphalt binder and mixture were investigated through a dynamic shear rheometer and indirect tensile strength testing. The results from this laboratory study indicated that 1) the HL can be further grinded into sub nano-sized particles by the LA Abrasion machine, with the average HL decreasing to about 600 nanometers, a sub nano scale; 2) an increase of 10 percent in Tensile Strength Ratio (TSR) was observed for HMA incorporating the sub nano-sized HL over the HMA added with un-processed (control) HL; 3) the asphalt mastics containing HL in various sizes did not show much change in terms of phase angle and complex modulus from Dynamic Shear Rheometer (DSR) testing results.

Key words: Anti-stripping, HMA, Lime, Nano.

Introduction

Many additives are now introduced in hot-mix asphalt (HMA) to improve the physical, rheological, mechanical and other performance properties of the mixture. Adding hydrated lime (HL), for example, has been accepted as a common practice in the production of HMA in most parts of the USA to improve moisture resistance of the mixtures. Enhancing the moisture resistance of asphalt mixtures is critical to fulfilling the designated performance of asphalt pavements [1-3]. In addition, many research projects have proven that there are other benefits of adding HL to HMA mixtures. Those benefits include the improved properties of resisting rutting, fatigue and cracking [4-11]. In short, HL added to HMA mixtures works as both an anti-stripping agent and mineral filler in the mixtures, generally resulting in better pavement performance and elongated pavement life.

The literature expounds on the mechanisms of the improvement in the anti-stripping property of HMA through its interaction with asphalt binder and aggregates. The HL works in the HMA as an anti-stripping agent mainly because of the reaction between the lime and aggregates, in addition to the reaction between the lime and the asphalt binder. The former reaction enhances the bond between the asphalt binder and the aggregate, while in the latter reaction HL reacts with highly polar molecules to inhibit the formation of water-soluble soaps that cause stripping. The polar molecules will react with the lime and form an insoluble salt that no longer attracts water [12]. These reactions increase the resistance of the asphalt mixtures to moisture. On the other hand, the lime binder forms a stiff mastics that's filled in the void between the aggregates, resulting in a stronger asphalt structure. HMA mixtures filled with stiff mastics generally exhibit slower aging and higher resistance to rutting, fatigue, and low-temperature cracking.

The interaction of HL with asphalt binder is dependent on the type of base binder source, the way HL is introduced into the HMA mixture, and the percentage and quality of the HL. The reaction of HL with an asphalt binder changes the chemical composition depending on the base binder [4]. Introducing lime slurry into the aggregates differs from treating the aggregates with a dry lime in both the uniformity and content of the interaction. An optimum percentage of the HL is usually 1 to 2 percent by weight of the mix, or 10 to 20 percent by weight of the liquid asphalt binders. Many state departments of transportation (DOTs) require the HL for HMA to satisfy the ASTM C 1097, and the minimum passing rate for sieve No. 200 (0.075 mm) is 85%.

The particle size of HL will influence its interaction with the asphalt binder. The finer the HL, the more reaction between the HL and asphalt and/or aggregate will be expected. On the other hand, a finer filler in the mastics works more like a liquid than a solid. Nevertheless, there are not many reports in the literature about the size effect of HL on the properties of HMA and asphalt mastics. Moreover, most HL currently used for HMA is in micro scale.

Nanotechnology has been developed in many fields, including civil engineering infrastructural materials such as cement concrete and asphalt mixtures. Researchers try to improve the materials' properties by including nano-sized additives. For example, it has been observed in the laboratory that nano-TiO₂ and CeO₂ are good

Associate Professor, Department of Construction Management and Civil Engineering, Georgia Southern University, Statesboro, GA, 30458, USA.

Associate Professor, Department of Environmental and Civil Engineering, The University of Tennessee, Knoxville, TN, 37996-2010, USA.

³ Postdoctoral Research Associate, Department of Environmental and Civil Engineering, The University of Tennessee, Knoxville, TN, 37996-2010, USA.

⁴ Professor, Chongqing Jiaotong University, Chongqing, 400074,

Corresponding Author: E-mail jshen@georgiasouthern.edu Note: Submitted March 10, 2009; Revised May 19, 2010; Accepted December 9, 2010.

ultraviolet absorbing or shielding agents, depending on the percentages added [13]. An ultrafine material was used to improve the properties of asphalt mixtures. The use of the ultrafine particles significantly increases the complex modulus of asphalt mastics at high temperature, in comparison to mastics made with classical fillers [14]. Since the nano-sized particles exhibit much higher surface area than micro-sized particles, it is hypothesized that nano-sized hydrated lime may have much more reaction between the nano-sized lime and the binders, producing a much stronger bond between binder and the aggregates.

Objectives and Scope

The objectives of this research are as follows:

- to examine the possibility of making nano-sized HL through a commonly available mechanical method in a civil material laboratory;
- to study the size effect of nano-sized HL on the anti-stripping properties of HMA containing nano-sized HL;
- to investigate the size effect of nano-sized HL on the rheological properties of the asphalt mastic modified with the different sizes of the HL.

A commercially available HL that is currently used in the production of HMA in GA, USA, was selected for making nano-sized lime in the laboratory. An LA Abrasion machine was employed to produce nano-scale HL powders. A microscope was utilized for measuring the size of HL produced by the machine. Indirect tensile strength ratio (TSR) was selected to evaluate the moisture sensitivity for a dense-graded mixture containing produced HL. Dynamic Shear Rheometer (DSR) was used for testing the rheological properties of asphalt mastics with 20 percent in weight of the HL produced by the LA abrasion machine.

Materials Used and Test Methods

Laboratory Production of Sub Nano-Sized HL and Size Measurement Using a Microscope

An LA abrasion machine was selected and used in producing nano-sized HL because of its availability in a construction materials laboratory and for the purpose of its low cost and ease of use. This selection of the LA abrasion machine for the production of nano-scale HL is also confined by the lack of better resources. This method is neither recommended nor practical for a large scale production. Before this research, there was a concern over whether it is possible to produce nano-sized lime particles, and how fine in size the particles could be produced, using the machine in a construction materials laboratory. The process of producing a finer HL is thus a goal of this research. That is the reason the process is described in the paper as one of the objectives of this research.

An HL sample with a weight of 500 grams was placed into the LA Abrasion drum along with 12 standard steel balls. Five different levels of rotations (0, 250, 500, 1000, and 2500) were initially set for the LA Abrasion drum to determine the effectiveness of the grinding on the production of nano-sized HL. The HL samples from the drum after the prescribed rotations were taken out for size analysis through a microscope.

A microscope made in Germany was employed for the measurement of particle sizes. The procedure below was followed in preparing slides for the measurement:

- Slowly warm glycerine jelly in an oven until it is a uniform liquid. This step is necessary because glycerine jelly is solid at room temperature.
- 2. Add 10 mL of liquid glycerine jelly and 10 mL of vacuumed water to a 50 mL beaker.
- 3. Mix water and jelly with a stirrer for about 10 seconds or until solution is uniform.
- 4. Using the end of a dry stirrer, scoop out a small amount of HL from the sample, adding it to the solution prepared in step 3. Then stir the mixture for one minute.
- Using a dropper, apply two drops of the mixture to the center of a microscope slide.
- 6. Carefully place a slide cover over the liquid on the slide.
- 7. Place the slide on a slide warming tray and allow the slide to set for 24 hours so that all the water can be evaporated.
- 8. Once the slide has been allowed to set, place the slide on a microscope and adjust the microscope to an appropriate viewing power and light source for the best viewing settings.
- 9. Measure the dimensions of 60 random particles in unit by referring to the scale. The amplitude is 400. One unit is $2.5 \mu m$.
- 10. From this data, calculate the average particle size.

DSR Test for Rheological Properties of Asphalt Mastics

One conventional asphalt binder, PG 64-22, was used as the base binder. Tested samples for DSR were made by mixing the base binder with HLs produced using the LA abrasion machine at different rotations (0, 250, 500, 1000, 2500 rotations).

Asphalt mastics were made at one percent filler content by the weight of total aggregates such that the filler content equals the asphalt mixtures (excluding the common mineral filler content from the aggregate blend). The HL added in the asphalt mastics was 20 percent of the weight of the base binder. Asphalt binder and filler were hand mixed at 165°C on an electric heating plate. Asphalt mastic samples with 8 mm diameter and 2 mm height were used for DSR testing.

An Anton Paar Physica MCR 501 Rheometer was used to perform the DSR test on all asphalt mastic samples at three temperatures (15°C, 25°C, and 40°C) and at two frequencies (0.01 Hz and 25 Hz). The complex shear modulus and phase angle of asphalt binder and mastics were obtained from the DSR test.

Indirect Tensile Strength for Moisture Susceptibility of HMA

One dense graded hot mix asphalt (HMA) mixture with 12.5 mm nominal maximum aggregate size (NMAS) was used for the moisture susceptibility test. The asphalt binder PG 64-22 was also used in the HMA mixture. The aggregates consisted of 50 percent limestone D-Rock, 15 percent No. 10 screenings, 25 percent natural sand, and 10 percent manufactured sand. Their gradations and specific gravities are presented in Table 1.

The optimum asphalt content of the mixture was determined to be five percent with the Marshall design method. To investigate the

Table 1. Properties of Aggregates Used for the 12.5 mm HMA.

Sieve	Limestone	No.10	Natural	Manufactured
size	D-Rock	Screening	Sand	Sand
5/8"	100%	100%	100%	100%
1/2**	97%	100%	100%	100%
3/8"	70%	100%	100%	100%
#4	21%	92%	98%	99%
#8	7%	61%	93%	82%
#30	4%	29%	63%	28%
#50	3%	21%	13%	17%
#100	2.0%	20.0%	2.0%	9.0%
#200	1.8%	16.0%	1.0%	5.0%
$*G_{sb}$	2.524	2.424	2.501	2.476

Note: $*G_{sb}$ = Bulk Specific Gravity.

Table 2. HL Size of the HL Produced by LA Abrasion Machine (Unit: Microns).

Rotation	0	250	500	1000	2500
Average	1.38	1.35	0.66	0.61	0.60
STDEV	0.56	0.54	0.35	0.32	0.25

effect of the anti-stripping additives on the moisture damage of HMA mixtures, an additional one percent mineral additive by weight of total aggregates was added into the design mix. For the control mix, an additional one percent of No. 10 screenings passing No. 200 sieve was added into the design mix so that all the mixes had similar aggregate gradation and filler content. The HLs produced using 0, 500, and 2500 rotations were selected as anti-stripping additives. The moisture susceptibility test was conducted following the AASHTO T283 protocol. SGC-compacted specimens (150 mm diameter) were divided into two groups: the control group and the moisture conditioned group. Moisture conditioning starts with introducing moisture in the specimens within the saturation range of 55 percent and 80 percent. Then the specimens are placed in a freezer for a minimum of 15 hours at -18 ± 5 °C. The specimens are then placed in a hot water bath at 60 ± 0.5 °C for 24 ± 0.5 hours. The moisture conditioned specimens are ready for testing upon removal from the hot water bath. They are kept in a 25 ± 0.5 °C water bath for 40 ± 5 minutes. The air voids content was controlled within the range of $7 \pm 1\%$.

The indirect tensile strength of the control group and the moisture conditioned group were tested at 25°C and at the loading rate of 76.2 mm/min. The indirect tensile strength is calculated as follows:

$$S_t = \frac{2P_{ult}}{\pi t D} \tag{1}$$

where

 S_t = indirect tensile strength;

 P_{ult} = peak load;

t =thickness of specimen; and

D = diameter of specimen.

The moisture susceptibility of HMA is indicated by the tensile strength ratio (TSR) as follows:

$$TSR = \frac{S_{tm}}{S_{tc}}$$
 (2)

where

TSR = tensile strength ratio;

 $S_{\rm tm}$ = average tensile strength of the moisture conditioned group;

 S_{tc} = average tensile strength of the control group.

Results and Discussions

Production of Nano-sized HL in a Laboratory

Table 2 lists all the average particle sizes and deviations of the randomly selected 60 particles from the slides.

Results from Table 2 indicate that the average particle size of the HL decreased as the rotation number increased. A 250 rotation did not decrease so much in the particle size of the sample. When a rotation of 500 was applied, the average size reached 0.66 microns (660 nanometers). Further increases in the rotation number would not effectively decrease the particle size. The reason for this inefficiency can be attributed to the coarse surface of the steel ball and the inner wall of the drum of the LA Abrasion machine compared with the nano scale. Another reason is the extreme activity of nano-sized materials. The nano-sized particles may stick together again after absorbing moisture and $\rm CO_2$ in the air. It may be concluded that a sub-nano sized HL of about 600 nanometers can be effectively produced using this machine.

The deviations were relatively large compared to the average of the particle sizes. The large deviations indicate that the particle sizes of the HL measured via microscope were not uniform. This is mainly because the lime was randomly impacted by the steel balls in the LA abrasion drum, and the original HL was also a blend of different sizes. Some other mechanical methods have to be used in order to get a more uniform nano-sized HL. Figs. 1 and 2 show the photos of the HL samples with no rotation and 500 rotations under the microscope.

ITS and TSR of HMA

Table 3 presents the results from indirect tensile strength tests (IDT). The indirect tensile strength (ITS) values of the HMA in dry condition did not change so much for all the samples compared with the control sample and those using HL (0 rotation). However, the HMA in wet condition (after free thaw) increased significantly with the addition of HL. An increase of 28 percent in the wet ITS was observed by adding HL (0 rotation, average size 1.34 microns) into HMA. An even higher increase of 36 percent in the wet ITS was obtained by adding HL (500 rotations, average size 660 nanometers). The difference between the two increase rates in the ITS was due to the addition of HL with different particle sizes. This difference, calculated as eight percent in the wet ITS, can be attributed to an average particle size decrease from 1.34 to 0.66 microns in this study. When HMA used HL (2500 rotations), a much higher gain in the wet ITS was found. The increase rate of the wet ITS reached 49 percent compared to the control samples. In other words, there was 21 percent gain in the wet ITS because a nano size of HL was included to the HMA.

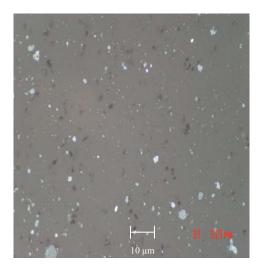


Fig. 1. HL Average Size: 1.38 Microns.

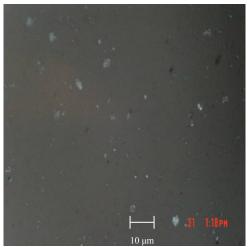


Fig. 2. HL Average Size: 600 Nano Meters.

Table 3. Indirect Tensile Strength (ITS) and TSR

	Dry ITS (psi)	Wet ITS (psi)	TSR
Control HMA	129.8	71.1	55%
HMA using HL (0 rotation)	128.1	91.4	71%
HMA using HL (500 rotations)	121.6	96.1	79%
HMA using HL (2500 rotations)	132.4	106.0	80%

The HMA had different wet ITS values for those using HL (500 rotations) and HL (2500 rotations) even though the average sizes of both fell in the same range of 600 nanometers. This difference is partially due to the fact that the latter has more nanometer particles since its deviation was smaller.

Overall, the benefit of using the sub nano-sized HL in improving the moisture resistance of HMA is obvious. The smaller the particle size used in HMA, the better the anti-stripping properties of the mixture. The reactions between the HL with the binder and the size used in HMA, the better the anti-stripping properties of the mixture.

The reactions between the HL with the binder and the aggregates will tend to go faster and more thoroughly. In this study, an increased rate of around 15 percent in the wet ITS value was obtained for a large nano size.

Similarly, the benefits of using the sub nano-sized HL on the TSR can be observed from Table 3. The TSR is a ratio representing the ITS of the samples from dry to wet. The significant increase of the ITS in wet conditions is a great contributor to the TSR. The TSR value of the HMA using the sub nano-sized HL increased an average of nine percent, compared to that using HL without rotations.

DSR of Asphalt Mastics

The results of the DRS test are listed in Table 4 for a frequency of 25 Hz and in Table 5 for a frequency of 0.001 HZ.

As expected, the addition of a filler increased the rheological properties. One percent of HL (no rotation) added to the mixture increased the complex modulus and decreased the phase angle. The added HL filler made the mastics stiffer and more elastic. The change responsible for the mastics to become stiffer and more elastic in the rheological properties is partially due to the reaction of the HL with the binder and partially due to the presence of HL particles in the binder. The finding was true for the testing conditions of three different temperatures at 15, 25 and 40°C and two frequencies of 25 Hz and 0.01 Hz.

The rheological properties did not alter so much when the asphalt mastics was added to the HL while rotated at different numbers in the LA abrasion machine. In general, the particle size of mineral filler affects the rheological properties. A fine particle of mineral filler works more like a liquid or extender in the mastics. At the same time, it will have more reaction with the binder. The two effects on the rheological of the mastics are conflicting. It suggests that the two effects in this study result in good balance, namely that there is neither an increase nor decrease in the phase angle and complex modulus.

Summary and Conclusions

The research in this paper aims to further improve the properties of HMA using smaller sized hydrated lime. A preliminary laboratory evaluation was conducted to study sub nano-sized HL on the selected properties of HMA. An LA Abrasion machine was selected for the production of smaller-sized HL, which is commercially available and currently used in the state of Georgia. The smaller particle sizes of the HL produced through different LA abrasion rotations were measured through a microscope. ITS and rheological properties were selected for the evaluation of the influence of HL particle sizes on moisture sensitivity of HMA mixtures and on rheological properties of asphalt mastic. Based on the results obtained, the following can be concluded:

Using an LA Abrasion machine available in a construction material laboratory, it is possible to produce finer sub nano-sized HL (down to 600 nanometers). The smallest possible average size of the HL was obtained when a rotation

Table 4. Rheological Properties of Asphalt Mastics Added with Different HL Obtained at 25 Hz.

Type of Asphalt Mastics	Frequency 25 (Hz)				
Type of Asphalt Mastics	Temperature (°C)	Phase Angle	Complex Modulus (kPa)	Storage Modulus	Loss Modulus
Base Asphalt Binder	15	42	346000	25733.3	23100
PG 64-22	25	56.7	86000	4720	7186.7
FG 64-22	40	70.6	15200	506.3	1430
Acabalt Dindon	15	39	557333.3	43300	35066.7
Asphalt Binder	25	54.5	146333.3	8506.7	11900
with 1% HL (0 rotation)	40	69.9	24900	851.3	2333.3
A subalt Dindon with 10/ III	15	39.3	546000	42266.7	34566.7
Asphalt Binder with 1% HL	25	54.8	138333.3	7973.3	11300
(250 rotations)	40	70.5	24033.3	803.3	2263.3
A subalt Dindon with 10/ III	15	39.1	567000	44000	35766.7
Asphalt Binder with 1% HL	25	55	145666.7	8343.3	11900
(500 rotations)	40	70.3	24966.7	841	2346.7
A sub-sit Dividen with 10/ III	15	39	557666.7	43300	35100
Asphalt Binder with 1% HL	25	54.7	141000	8166.7	11533.3
(1000 rotations)	40	71	24800	808	2343.3
A surbalt Dindon suith 10/ III	15	39.9	522666.7	40100	33533.3
Asphalt Binder with 1% HL	25	55.5	132333.3	7486.7	10866.7
(2500 rotations)	40	69.6	22800	796	2136.7

Table 5. Rheological Properties of Asphalt Mastics Added with Different HL Obtained at 0.01 Hz.

Type of Ambalt Mastics	Frequency 0.01 (Hz)				
Type of Asphalt Mastics	Temperature (°C)	Phase Angle	Complex Modulus (kPa)	Storage Modulus	Loss Modulus
	15	74.2	2860	78.1	275.7
Base Asphalt Binder PG 64-22	25	82.7	196.3	2.5	19.5
	40	87.5	7.3	0	0.7
A amb alt Dindon with 10/ III	15	73.1	5253.3	152.3	502.7
Asphalt Binder with 1% HL	25	82.1	360.7	5	35.7
(0 rotation)	40	87.1	13.3	0.1	1.3
Applied Dinden with 10/ III	15	73.1	5090	148	487
Asphalt Binder with 1% HL	25	82	354.3	5	35.1
(250 rotations)	40	87	13.2	0.1	1.3
Asabalt Dindon with 10/ III	15	73.1	5296.7	154	506.7
Asphalt Binder with 1% HL	25	81.9	373.7	5.3	37
(500 rotations)	40	86.2	13.6	0.1	1.4
Applied Dieden with 10/ III	15	72.9	5256.7	154.3	502.3
Asphalt Binder with 1% HL	25	81.7	366.3	5.3	36.2
(1000 rotations)	40	88.3	13.1	0	1.3
Asphalt Dindon with 10/ III	15	73.2	4733.3	136.3	453.7
Asphalt Binder with 1% HL	25	82	329.7	4.6	32.6
(2500 rotations)	40	85.9	12	0.1	1.2

of 500 (or more) is set for the machine. Further increase in the rotation did not effectively decrease the average size of the HL. This is partially due to the coarse surfaces of the steel balls and the inner wall of the drum of the LA Abrasion machine. In addition, sub nano-sized HL is more active due to its high surface area, which makes it prone to stick together again once exposed to air.

- The smallest average size of the HL produced by the LA abrasion machine was about 600 nanometers, measured from a microscope. The deviation of the average size was relatively large, indicating that the particles were not uniformly grinded
- using LA Abrasion steel balls. In order to obtain a uniform nano-sized HL, other grinding methods have to be used.
- 3. The influence of sub nano-sized HL on improving the anti-stripping of HMA is significant. Generally, small-sized HL increases wet ITS and TSR values as compared to conventional HL treated HMA. An increase of 15 percent in ITS and eight percent in TSR were observed for a dense graded HMA using HL with an average size of 600 nanometers compared with those using HL without rotation.
- 4. The rheological properties of the mastics, with the inclusion of HL in different sizes, were improved greatly compared with those of the controlled sample. This finding is consistent with

- other research. With the decrease of the HL particle size, the phase angle and complex modulus varied insignificantly.
- The current suggestion is that a study on the properties of HMA using nano-sized HL be conducted.

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