

Cellulose Nanocrystal Modified Bitumen Pavement Materials

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ABSTRACT

The objective of this research was to assess the potential of cellulose nanocrystals (CNCs) at laboratory scale within paving materials that make use of bituminous binders. Brittleness potential as measured by AASHTO T401 protocols was the primary evaluation mechanism where unconditioned, laboratory-conditioned, and field-aged materials were assessed. The investigation was divided into two components, neither of which are prevalent in literature or practice. The first evaluated CNCs within traditional dense graded asphalt mixed at temperatures on the order of 160°C and the second evaluated emulsion-stabilized sand at mixing temperatures on the order of 65°C. The emulsion-stabilized sand was the main focus as it was used to evaluate CNCs' potential in pavement base layers. The primary conclusion was that emulsion-stabilized sand observed drastic behavior improvements at some dosages across a variety of treatments ranging from unaged, to combined effects laboratory conditioning, to field aging.

INTRODUCTION AND BACKGROUND

This paper presents findings of a pilot scale investigation of cellulose nanocrystals (CNCs) used to modify pavement materials with bituminous binders. Cellulose is the most abundant natural polymer available on Earth with its main sources being wood pulp and cotton fibers. CNCs are an emerging material with considerable possibility for many applications, construction materials being one candidate. The objective of this research was to assess the potential of CNCs at laboratory scale within paving materials that made use of bituminous binders. The focus of the work was a series of experiments with various additives to asphalt. The results showed considerable laboratory scale promise of CNCs to modify emulsified asphalt and effectively stabilize sandy materials. This paper also documents how the CNCs were introduced into the emulsion stabilized sandy materials. Brittleness potential as measured by AASHTO T401 (i.e. Cantabro Mass Loss) protocols was the primary evaluation mechanism where unconditioned (virgin) materials, laboratory conditioned materials, and field aged materials were assessed. Laboratory conditioning was intended to simulate a few years of field aging, and the experimental matrix also included up to two years of actual field aging in a hot and non-freeze climate. Cracking and brittleness potential are one of the most formidable challenges being faced by the modern paving industry (e.g. Howard et al. 2016), and as a result, emphasis was placed herein on reducing brittleness potential.

The CNC investigation was divided into two components, neither of which are prevalent in literature or practice. The first evaluated their use in traditional dense graded asphalt (DGA) at

temperatures on the order of 160 °C. The second evaluated CNCs within emulsion stabilized sand (ESS) mixed at temperatures on the order of 65 °C. The first evaluation (i.e., traditional plant mixed asphalt) is a larger market, but the mixing temperatures are challenging for a cellulose based material. The second evaluation (i.e. emulsion stabilized sands or sandy materials) is a smaller market, but still encompasses many non-surface paving layers such as emulsion stabilized bases, in-place recycled bases, and cold-central-plant recycled bases. The majority of this paper is devoted to ESS and base layers of pavements.

LITERATURE REVIEW

CNCs have been studied a considerable amount in concrete (e.g. Cao et al. 2015), with some promising results when the binders used are hydraulic (e.g. portland cement, portland-limestone cement, fly ash, slag cement). As an example, Fu et al. (2017) reported CNCs improving degree of hydration and flexural strength by up to 20% in cement pastes.

Temperature susceptibility is an obstacle to overcome for many paving applications. Molnes et al. (2016) reviewed several works and reported that CNCs break down above 220 °C; experiments were performed on dried CNCs with an inert atmosphere of nitrogen gas. Interactions with CNC experts at the United States Department of Agriculture (USDA), United States Forest Service, Forest Products Laboratory (FPL) revealed the following. High lignin CNCs, as of the 2018 time frame, were indicating a degradation temperature (short duration) in thermogravimetric analysis (TGA) of roughly 300 °C. For extended durations, this temperature was expected to decrease in the view of FPL experts. CNCs were noted to thermally degrade above around 160 °C (320 °F), but coating in ethylene glycol might be helpful for reducing temperature susceptibility.

Li et al. (2017) estimated cellulose and its derivatives as the most abundant renewable organic materials in the biosphere with annual production estimated to be over $7.5(10^{10})$ tons. Cellulose is often present in plant cell walls and can also be found in marine animals, bacteria, fungi, and algae. Bulk cellulose consists of two regions; 1) highly ordered regions, and 2) amorphous regions. When subjected to treatment, the highly ordered regions can be extracted to result in the collection of CNCs (George and Sabapathi 2015).

Current applications of CNCs include products such as pH sensors, Pickering emulsions (i.e. emulsions that are stabilized by solid particles), and reinforcing agents for polymer nanocomposites (George and Sabapathi 2015). Challenges that have limited industrial use include prediction of long-term properties and issues with water sorption as well as concerns with thermal stability, durability, and safety (Brinchi et al. 2013). CNC's can bio-degrade under appropriate environmental conditions.

CNCs can be dispersed in a variety of materials including water, dimethyl sulfoxide, and acids such as hydrogen chloride. Following acid hydrolysis, CNCs are often diluted with water and then dialysis against the water is performed to remove free acid and sugar molecules (Habibi et al. 2010). CNC particles are isolated from water in numerous ways including air-drying, freeze-drying, or spray-drying. Each drying method produces a different CNC product. When left undisturbed to air-dry, the resulting CNCs form a solid but brittle film that is glossy and iridescent. Freeze-dried CNCs usually result in a product made up of thin lamellar flakes while spray-dried CNCs typically result in a free-flowing and flour-like powder (Beck et al. 2012).

Rheological behavior of CNCs is concentration dependent. At high concentrations they exhibit an elastic gel-like behavior while at low concentrations they exhibit viscous liquid like behavior (Li et al. 2015). CNCs show shear thinning behavior at lower concentrations (George

and Sabapathi 2015). CNCs have high aspect ratios, high mechanical properties, low thermal expansion, low density, and surface-accessible hydroxyl groups that can readily be chemically modified to give additional functionalities (Moon et al. 2016).

CELLULOSE NANOCRYSTAL MODIFIED PLANT MIXED ASPHALT

Twelve dense graded asphalt (DGA) mixes were produced with a range of components including reclaimed asphalt pavement (RAP), recycled asphalt shingles (RAS), virgin aggregates (gravel, limestone, sand), and virgin binder contents from 4.3 to 7.5%. All mixes had 9.5 mm nominal maximum aggregate size (NMAS) gradations, were mixed at 163 °C, and were compacted at 149 °C. PG 67-22 asphalt binder was used throughout the DGA experiments. Up to 1% freeze-dried (FD) CNC powder by binder mass was evaluated alongside crude tall oil (CTO) at up to 5% binder mass. CTO is a base for several bio-based rejuvenating products, which was the rationale for inclusion in this work and in particular to evaluate any possible synergies with CTO and FD CNCs. The CNCs evaluated with DGA was different than the one evaluated with emulsions.

Materials. The CNCs were high lignin CNCs supplied by the FPL in a freeze-dried cake. This product is referred to as FD CNCs. The material was extracted from medium-density fiberboard at 185°C for 180 minutes. In its solid, freeze-dried state it is dark brown in color and can easily be broken apart into a fine powder for use. PG 67-22 asphalt binder was sourced from Ergon. The CTO was an acidulated product from sulfate black liquor skimmings containing straight chain C18 and cyclic C20 organic acids. At 25 °C, CTO was a liquid with a specific gravity of roughly 0.97 g/cm³. The RAP had a continuous grade of PG 105-7 and an average binder content of 4.6%. The RAS had a continuous grade of PG 209+24 and an average bitumen content of 17.1%.

Mass Loss Testing. Figure 1 summarizes AASHTO T401 Mass Loss (ML) test results from these twelve mixes in equality plots; full data is available in a sponsor report (Howard et al. 2024). Figure 1 includes unaged, laboratory conditioned, and field aged data that was exposed to the same conditions as the ESS described in the next section. Lower ML values are desired and indicate mixes that have less brittleness potential; slopes less than 1.00 for regression through the origin suggest an additive(s) outperformed control mixtures.

Mixtures without additives outperformed mixtures with additives. FD CNCs had a slope of 1.02, which indicates a slight increase in mass loss due to their addition. Given the nature of the experiments performed, a slope this close to 1.00 would, overall, suggest that FD CNCs performed in a manner comparable to, perhaps slightly worse than, control mixtures without additives. When CTO was added, performance decreased sharply. A slope of 1.13 is a fairly clear indication that the CTO, as added to these mixtures, had a negative impact on mechanical behavior. Interestingly, the combination of FD and CTO had a slope of 1.16, which is almost identical to the sum of the increase in slopes of the two materials individually relative to the control (i.e. $0.02 + 0.13 = 0.15$). For purposes of this paper, FD CNCs in traditional plant mixed asphalt seemed neutral, overall, and as such most emphasis in this paper is placed on ESS in the next section.

CELLULOSE NANOCRYSTAL MODIFIED EMULSION STABILIZED SAND

Three emulsion stabilized sand (ESS) mixtures were produced and are denoted M0, M1.1, and M2.2 herein. For reference, these mixtures are denoted M47, M48, and M49, respectively, in the long term aging experiment described later in this section.

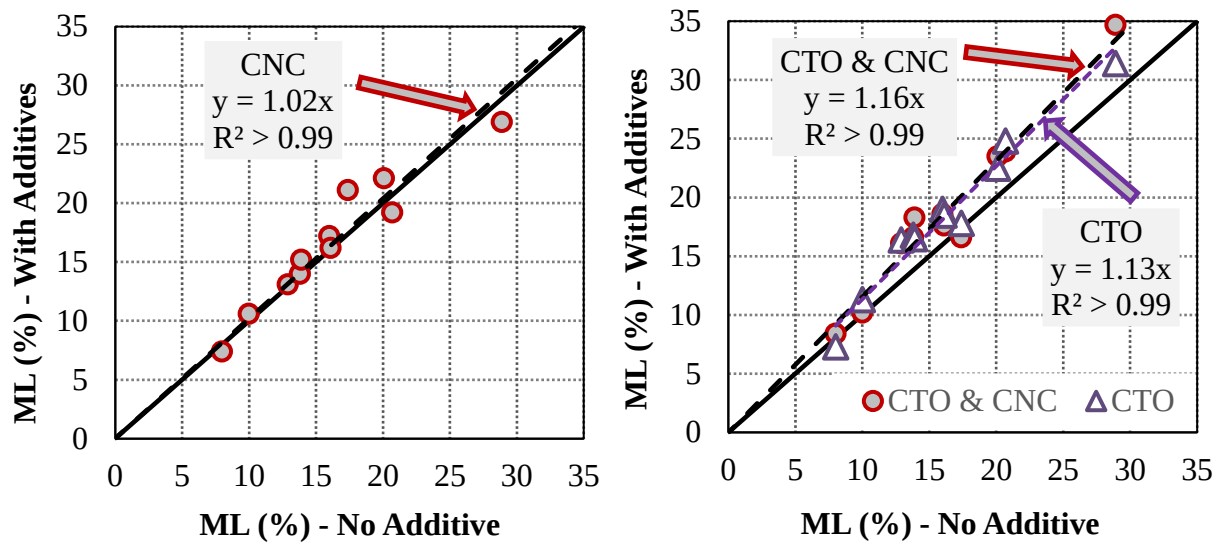


Figure 1. Summary of Plant Mixed Asphalt Behavior Containing Cellulose Nanocrystals

Materials. Water dispersed CNCs, referred hereafter as aqueous CNCs, were provided in a gel (aqueous) form suspended in water at 10.7% by weight. The gel was an opaque viscous liquid with a bulk density of about 1 g/cm^3 , a neutral pH, and 1% by weight sulfur residual (dry CNC mass) in sodium form. A CRS-2P emulsion, supplied by Ergon, with a 68% residue was used in conjunction with sand having a bulk specific gravity of 2.560 and a water absorption of 1.2 wt.%. The sand's gradation was (percent passing); No. 4 of 99%, No. 8 of 82%, No. 30 of 57%, No. 50 of 16%, No. 100 of 1%, and No. 200 of 0.1 wt.%.

Preliminary Testing for Mixture Proportioning. To the author's knowledge, the efforts in this research were the first attempts to mix CNCs with asphalt emulsion and sand. To gage proportions, two preliminary cases were evaluated. Case 1 made use of 5,000 grams of sand, 150 grams of emulsion (102 grams of bituminous residue), 150 grams of water, and 0 grams of aqueous CNC. Case 2 made use of 5,000 grams of sand, 150 grams of emulsion (102 grams of bituminous residue), 75 grams of water, and 75 grams of aqueous CNC (8.25 grams of residual CNC). Case 2 had a CNC dosage of roughly 8.1%, and as seen in Figure 2, this amount of CNC with only 102 grams of bituminous residue did not produce a stable specimen.



Figure 2. Preliminary ESS Proportioning Exercises

The Case 1 specimen held its shape, but there was noticeable lack of sand coating. These experiments were performed where all ingredients were heated to 150 °F, mixed, and immediately compacted with 30 gyrations of a Superpave Gyratory Compactor (SGC). All mixing tools, the mixing bucket, and the compaction mold were at room temperature. The findings from these preliminary experiments led to an increase in emulsion content, a decrease in CNC's content, and pre-coating the sand with CNC's as described in the next section.

Incorporating Aqueous CNC Materials Into Mixes. Aqueous CNCs were incorporated into ESS mixtures by diluting them with water before coating sand with the mixture while all ingredients were at 65°C (150 °F). To enhance its coating capabilities, additional water was mixed with the aqueous CNCs in a ratio of 2:1 (i.e. two parts water to one part aqueous CNCs) and then heated to 65°C in a water bath. The liquid mixture was added to the sand in grams of aqueous CNCs per kg of sand in ratios of 5:1 and 10:1 to produce mixtures with approximately 1.1 and 2.2% aqueous CNC residue within the asphalt emulsion residue (actual CNC dosages are shown later). Preheated sand was treated in individual 5 kg batches by means of a 5-gallon mixing bucket and then air dried to a constant mass in pans prior to heating and mixing with emulsion.

For example, M1.1 had about 1.1% CNC and was prepared by combining 25 g of the original aqueous CNC with 50 g of water in a metal tin. The tin was then covered and placed in a 65°C water bath until it reached mixing temperature. Thereafter, 5 kg of 65°C preheated sand was placed in a mixing bucket and the heated aqueous CNC/water combination was mixed into the sand until all sand particles were dampened. The coated sand was then placed into a pan and set aside to dry for several days. At the conclusion of mixing sand and aqueous CNC, 25 g (in this example for M1.1) or 50 g (in the case of M2.2) of CNC residual coated 5 kg of sand. With an original aqueous CNC moisture content of roughly 89%, approximately 2.75 grams (M1.1) or 5.5 grams (M2.2) of CNC residue remained after drying. A total of 368 grams of emulsion would be added to this 5 kg of sand, where the residue value was approximately 68%, or 250 grams of residual binder and 2.75 grams of residual CNC material was roughly 1.1% of the residual emulsion bitumen.

Specimen Mixing, Compaction, and Proportions. ESS mixtures were mechanically mixed in a bucket and compacted at 65°C (150 °F). Emulsion and sand pre-coated with CNCs were heated to 65 °C, mixed, and then compacted to 125 gyrations with an SGC with no short term aging period. All mixing tools and molds were at room temperature. Bulk specific gravity was measured in accordance with AASHTO T331 (vacuum sealing) to obtain air void (V_a) values when maximum mixture specific gravity was measured according to AASHTO T209. Binder content (P_b) on a mixture mass basis was 4.8% for all experiments. A total of 368 grams of emulsion (250 g of residue) was added to 5,000 g of sand containing 0, 2.75, or 5.5 g of CNC residue for a total mixture mass of 5,250 to 5,256 g. The three dosages of CNC were: M0 at 0% (0 g of residue), M1.1 at 1.1% (2.75 g of residue), and M2.2 at 2.2% (5.5 g of residue).

Laboratory Conditioning and Field Aging. Specimens were handled prior to testing in one of three manners: unaged, laboratory conditioned, or field aged. Unaged specimens were produced, stored in ambient air conditioned laboratory conditions away from direct sunlight to represent properties immediately after being placed on the roadway. In their unaged state, emulsion stabilized materials are generally the most resistant to raveling, cracking, and otherwise most resistant to weathering, but they are also generally the least resistant to rutting under traffic.

One conditioning protocol (CP) that was presented in Smith and Howard (2019) was used in this study; this protocol is often referred to as CP7. Three conditioning mechanisms were used to

simulate aging: oven conditioning at 85°C for 5 days, hot water bath conditioning at 64°C, and a freeze-thaw (FT) cycle. Room temperature specimens were placed in a preheated force-draft oven and left to age for 120 ± 0.5 hours. After conditioning was completed, specimens were cooled to room temperature with the oven turned off and the door opened. Specimens were then vacuum saturated within a range of 70 to 80% of AASHTO T166 measured V_a volume. Specimens were then stored in submerged room temperature water before being transferred to a 64°C preheated water bath for hot water conditioning for 14 days. When hot water conditioning was completed, specimens were again cooled to room temperature while still submerged in water. Following hot water conditioning was a single FT cycle at -22°C. Specimens were transferred directly from the room temperature water to a prechilled freezer for 24 hours. The freezer was then turned off and specimens were thawed. Thereafter, specimens air dried for at least 42 days prior to testing.

Field aging occurred in Columbus, MS for 1 year (1F) or 2 years (2F). This aging site has been used for over a decade as of this report's date and has aged 63 different mixtures. Figure 3 provides representative photos of ESS aging, and Smith et al. (2019) summarizes representative conditions at this site as measured by a nearby weather station. All specimens were aged in PVC sleeves so that only specimen tops were subjected to direct sunlight and weathering.

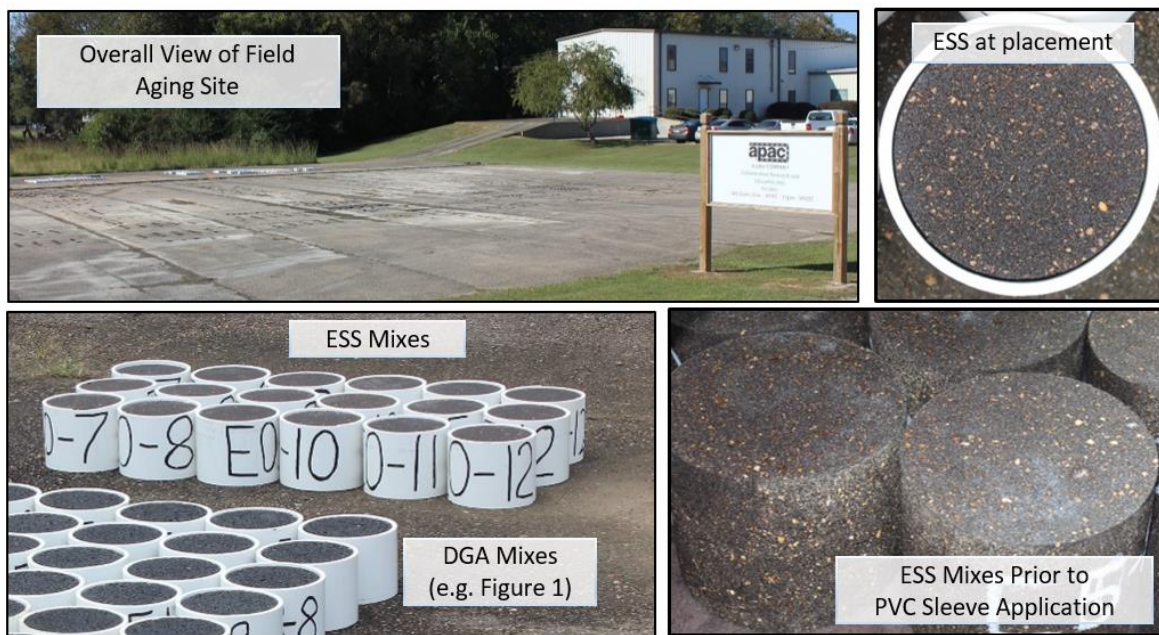


Figure 3. Field Aging Site and Specimens

Mechanical Property Testing. Cantabro Mass Loss (CML) testing was performed according to AASHTO T401. A compacted asphalt specimen is placed into an LA Abrasion drum absent steel spheres at 25 °C and is subjected to 300 drum revolutions. Mass loss (ML) is the change in specimens mass divided by the original mass reported as a percentage. Cox et al. (2017) and Doyle and Howard (2011) provide more information on CML testing of compacted asphalt.

Test Results. Tables 1 and 2 summarize ESS test results. Final height (Table 1) and air voids data for these mixes suggest the CNC's modestly inhibited compaction as the compacted height

increased as a function of CNC content. V_a measured on these specimens supported the height observations as the average air voids for M0 were 12.4% (Stdev of 0.7%), while the average air voids for M1.1 and M2.2 were 13.0% (Stdev of 0.6%) and 13.0% (Stdev of 0.8%), respectively.

When compared to the control mixture, 1.1% CNC dosage behaved in a comparable manner (Table 2). However, doubling the dosage to 2.2% led to fairly drastic behavior improvements for all treatments. Quantification of the benefits of the 2.2% CNC dosage is not feasible from these ESS mixes, but the potential benefits to performance are very clear given the large reduction in mass loss observed. The behavior differences between M0 and M2.2 are the most impactful finding in this paper. The magnitude of difference in T401 results shown between M0 and M2.2 exceed some of the most impactful findings of Cox et al. (2017) that was a formidable step towards adoption of T401.

Table 1. Gyratory Test Results

Mix ID	n	Compacted Height (mm)		
		Minimum	Maximum	Average
M0	12	126.7	130.1	128.0
M1.1	12	126.7	129.0	128.3
M2.2	12	126.9	129.9	128.6

Table 2. AASHTO T401 Test Results

Mix	ML (%)			
	Unaged	CP7	1F	2F
M0	11.2	81.0	11.9	17.3
M1.1	12.8	76.6	13.1	14.1
M2.2	4.9	43.8	9.7	11.2

-- Each value is the average of three replicates.

One note of relevance is that CP7 conditioning meaningfully damaged these sand asphalt specimens (Figure 4). All nine specimens were able to be tested, but there were large and obvious cracks within several of the specimens. These cracks likely affected the magnitude of the values measured and reported, but any damage during the protocol is realistic of damage that could occur in service and all mixtures received identical treatment.



Figure 4. Visual Damage to Sand Asphalt During CP7 Conditioning

DISCUSSION

The results presented in the previous section clearly show a potential for performance improvement (less brittle) for CNCs as evaluated in laboratory conditions. These property improvements, however, should be considered in conjunction with a few additional factors. One factor is work in a new sector can emerge over a period of many years and earlier works don't often attempt to address all major factors for implementation in earlier stages. The emergence of microbially induced calcite precipitation (MICP) to stabilize soils is one example. Choi et al. (2020) is a review paper that shows the progression of MICP over several years and even in present day, there remain numerous challenges for full-scale and widespread implementation. Similarly, Fatehi et al. (2021) is a review paper on biopolymers for soil stabilization that shows a similar progression over an extended period of time. CNCs within emulsions used to stabilize soil or aggregates should be viewed in the infancy of a time scale comparable to MICP or biopolymers.

A second factor to consider is economics. As of August of 2023, \$22/kg (\$10/pound) was a reasonable estimate for CNC. Recent data from Baumgardner et al. (2021) had SBS polymer (a commonly used asphalt binder additive) at 2.51/kg (\$1.14/pound), but during supply shortages prices were documented to rise to \$4.41/kg (\$2.00/pound). A typical dosage rate for SBS to modify asphalt binder is 3% (i.e. 60 pounds of SBS and 1,940 pounds of bitumen producing one short ton of product). On this same basis, M2.2 would be 43 pounds of CNC and 1,957 pounds of bitumen producing one short ton of product. At a typical price, CNCs would cost 3.5 to 6 times more than SBS for the aforementioned ranges of prices and conditions. Other forms of cellulosic nanomaterials are options that have much lower price expectations. One is mechanical grade cellulose nanofibrils that contains lignin. An estimated commercial volume price for this material is on the order of \$0.50/pound, a more suitable price point for this application. As of yet they have not been evaluated in asphalts.

A third factor is production at an actual facility. Pre-coating aggregates with CNCs as occurred in this effort is likely not cost effective or efficient at full-scale. Introduction of the CNCs into emulsion is likely the most efficient, and if this occurs, any performance differences from a new introduction mechanism should be considered. A fourth factor would be to evaluate a wider range of gradations in the presence of CNCs. A clean sand was used herein to establish a baseline of properties. A fifth and final consideration would be to measure a wider range of characteristics beyond brittleness to assess CNCs for paving materials.

SUMMARY AND CONCLUSIONS

This research assessed cellulose nanocrystals (CNCs) in paving materials making use of emulsified asphalt, and emphasized pavement base layer type applications in a laboratory scale effort. This study aligns conceptually with efforts in recent years looking at emerging techniques to better stabilize soil and aggregates such as biopolymers and bacterially derived cements that are considerably different from more historical and traditional approaches. The primary conclusion of this effort is that emulsion stabilized sand observed drastic behavior improvements at some dosage rates across a range of treatments ranging from unaged, to combined effects laboratory conditioning, to field aging. The behavior of emulsion stabilized sand warrants further consideration of the performance potential of CNCs for paving applications where mixing temperatures are on the order of 65 °C or less. Economic challenges are noted and were briefly

discussed earlier in the paper. Use of FD CNCs containing lignin in plant mixed asphalt at temperatures on the order of 160 °C did not show promise as they performed in a manner comparable to, perhaps slightly worse than, control mixtures without additives.

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