

**Project Title**

Characterization of the Plant-Based Bio-Asphalt Binder and Bio-Additives

**University**

South Dakota State University

**Principal Investigator**

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**Research Needs**

It is estimated that more than 27 million tons of asphalt binder with a value of over 12 billion dollars are annually used in the construction of U.S. highways. Asphalt binder used for pavement construction is obtained mainly by the distillation of crude oil in refineries. The scarcity of natural resources, environmental concerns, and emerging needs for sustainable materials have spurred development and use of materials and processes that are renewable and environmentally friendly. In response to this need, initiatives have been taken to develop a new generation of bio-based construction materials, as a result of continuous innovation in the use of agricultural products, byproducts and biomass as a material feedstock. Development of plant-based bio-asphalt binder to replace the petroleum-based binder is a major element of these initiatives. Therefore, development and evaluation of innovative plant-based asphalt binders and additives, such as cellulose and lignin will help to increase use of bio-materials to maximize the sustainability of ground transportation system. The proposed study aims at evaluating bio-asphalt binders and additives derived from agricultural products and byproducts prevailing in North Central Region (e.g., corn and soybean) as the primary feedstock. This will be achieved through a laboratory testing program to evaluate the performance of asphalt mixes containing plant-based bio-asphalt binder and cellulose Nano-fibers. The results of this research will allow the research team collect the necessary data to assess the feasibility of using these materials in construction of asphalt pavements in South Dakota and elsewhere. Also, the proposed project will help the research team enhance the existing research infrastructure at SDSU's asphalt laboratory.

**Research Objectives**

The main objective of the proposed study is to design and produce asphalt mixes using plant-based bio-asphalt binder and evaluate their performance through an experimental laboratory testing program. Also, the feasibility of using cellulose Nano-fiber as an asphalt additive will be studied through a laboratory performance testing of asphalt mixes containing this additive. The specific objectives of the proposed study are as follows:

1. Design and produce asphalt mixes containing different amounts of plant-based bio-asphalt binder and cellulose Nano-fibers in the laboratory;
2. Evaluate the most important pavement performance measures of the designed and produced bio-asphalt mixes and asphalt mixes modified by using cellulose Nano-fibers in the laboratory through a comprehensive testing program. The proposed pavement performance measures to be tested consist of rutting, fatigue cracking, and moisture-induced damage potential;
3. Determine dynamic modulus, as an important M-EPDG input parameter of the bio-asphalt mixes and asphalt mixes modified by using cellulose Nano-fibers in the laboratory;
4. Establish and optimize the best practice for the laboratory-scale production of cellulose Nano-fibers for transportation and pavement applications using electrospinning method;
5. Collect the necessary data to assess the feasibility of using these materials in construction of asphalt pavements in South Dakota.

## Research Methods

Bio-asphalt binders can be used in three categories as: (1) binder replacement (100% bio-binder); (2) asphalt extender (25% to 75% bio-binder); and (3) binder modifier (less than 10% bio-binder) (Brown et al., 2009). In addition, engineered asphalt binders are modified by using different petroleum-based polymers as binder additives, to enhance their mechanical properties. In view of polymers' non-renewable source and high price, they are considered as unsustainable materials. Hence, replacement of the polymers with cellulose Nano-fibers will result in cost-effective and sustainable asphalt mixes. Therefore, the proposed research will focus on (1) evaluating the performance of the asphalt mixes designed and produced by using plant-based bio-binders and cellulose Nano-fibers; (2) laboratory production of cellulose Nano-fibers; and (3) characterization of the lab-produced cellulose Nano-fibers. For this purpose, the required materials for this study, namely bio-fuel production waste, petroleum-based and plant-based bio-asphalt binders, and graded aggregates will be collected. The cellulose will be extracted from bio-fuel waste using alkali and enzymes. The extracted cellulose fiber will be characterized and its properties will be correlated with binder outcomes. Specifically, laser scanning microscopy (LSM) images will be used to characterize the fiber size and morphological features. The composition and degree of crystallinity will be characterized using X-Ray Fluorescence (XRF) and Raman spectroscopy. Finally, the stiffness of cellulose fiber will be obtained using Nano-indentation (NID). Workflow proposed for production and characterization of the cellulose Nano-fiber is presented in Figure 1.

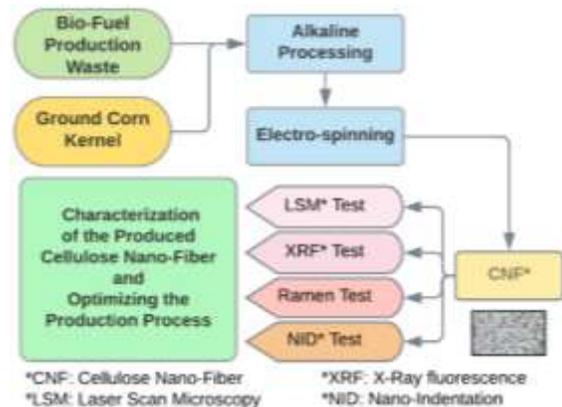


Figure 1. Workflow for laboratory-scale production and characterization of cellulose Nano-fiber

In addition to characterization of the cellulose Nano-fibers, laboratory tests will be conducted to evaluate four important performance measures of asphalt mixes. The workflow and test matrix proposed for testing asphalt binders and mixes are

presented in Figure 2. For this purpose, a PG 58-28 asphalt binder will be mixed with 2, 5, and 7% of the cellulose Nano-fiber. Also, the collected plant-based bio-asphalt binder will be mixed with PG 58-28 asphalt binder with amounts of 0, 7, 25, 50 and 100% by the weight of binder. These binder mixes will be used to design and produce 8 different types of Superpave® asphalt mixes, namely HMA-1 through 8. The produced asphalt mixes will be compacted to  $7.0 \pm 0.5\%$  air voids in a Superpave® gyratory compactor (SGC). In order to characterize the resistance of asphalt mixes to fatigue cracking and rutting, semi-circular bend (SCB) and flow number (FN) tests will be conducted on HMA samples using an asphalt standard tester according to AASHTO TP 105-13 and AASHTO T 378-17 standard test methods, respectively. Hamburg wheel tracking (HWT) and tensile strength ratio (TSR) tests will also be conducted on asphalt samples to evaluate their resistance to rutting and moisture damage according to AASHTO T 324-17 and AASHTO T 283, standard test methods, respectively. Additionally, dynamic modulus (DM) test will be conducted on asphalt mixes in accordance with AASHTO T 378-17 to determine DM values as an important M-EPDG design parameter of the bio-asphalt mixes. Finally, the optimum plant mixing temperatures for asphalt binder mixes will be determined by conducting the rotational viscometer (RV) test, according to AASHTO T 316-13 test method. After completion of the testing program, findings of this study will be compiled and analyzed. Important enhancements in the performance of the asphalt mixes containing plant-based bio-binder and cellulose Nano-fibers will be summarized and reported.

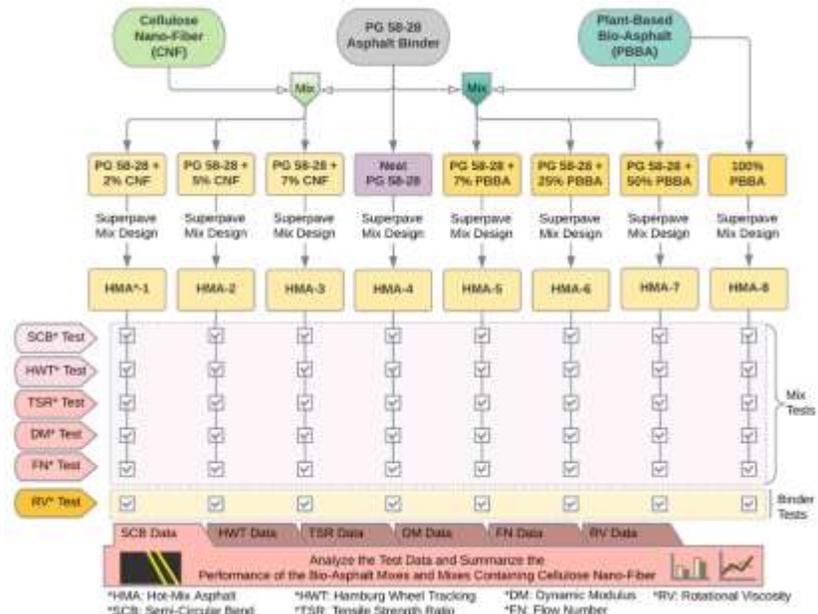


Figure 2. Workflow for performance evaluation of asphalt mixes containing plant-based bio-binder and cellulose Nano-fiber

Hamburg wheel tracking (HWT) and tensile strength ratio (TSR) tests will also be conducted on asphalt samples to evaluate their resistance to rutting and moisture damage according to AASHTO T 324-17 and AASHTO T 283, standard test methods, respectively. Additionally, dynamic modulus (DM) test will be conducted on asphalt mixes in accordance with AASHTO T 378-17 to determine DM values as an important M-EPDG design parameter of the bio-asphalt mixes. Finally, the optimum plant mixing temperatures for asphalt binder mixes will be determined by conducting the rotational viscometer (RV) test, according to AASHTO T 316-13 test method. After completion of the testing program, findings of this study will be compiled and analyzed. Important enhancements in the performance of the asphalt mixes containing plant-based bio-binder and cellulose Nano-fibers will be summarized and reported.

## Expected Outcomes

Specific outcomes and impacts of the proposed study are listed below:

- The proposed project is expected to promote use of the sustainable bio-materials and agricultural byproducts as the main feedstock for production of bio-asphalt binder. This will result in a reduced need for petroleum-based binders, which leads to numerous environmental benefits. In addition, due to availability of the bio-materials and their lower cost, a significant saving in asphalt binder cost is projected as a result of this project. Therefore, the economic and environmental impacts of the proposed project are expected to be significant by any measure.
- Currently, a majority of asphalt mix designs are conducted by using traditional petroleum-based asphalt binders. In the proposed study, asphalt mixes containing different amounts

of plant-based bio-asphalt binder and cellulose Nano-fibers will be designed in the laboratory. This will provide important information regarding the limitations and advantages of designing mixes using bio-asphalt binders and additives.

- The rutting, fatigue cracking, and moisture-induced damage potential database developed through laboratory testing of bio-asphalt mixes and asphalt mixes modified by using cellulose Nano-fibers will help pavement engineers gain a better understanding of the performance of asphalt mixes containing bio-asphalt binder. Use of the acquired knowledge in design and construction will contribute to durability and longevity of the asphalt pavements constructed by using bio-asphalt binders.
- Evaluation of asphalt mixes containing bio-asphalt binders and additives will increase industry confidence in using bio-materials, making construction more affordable (reduced cost due to reducing use of petroleum-based binder) and environment friendly – an important element of sustainable transportation infrastructure. It is also a major MPC goal (Economic Competitiveness).
- The proposed study will advance the workforce development goal of the MPC through providing important experiential learning opportunity to graduate research assistants (GRAs) and undergraduate students, to be appointed on this project. Experience suggests that GRAs are more likely to pursue career in transportation.
- In view of the timeliness of the research on bio-asphalt and bio-additives, it is expected that the findings of this study and acquired data will significantly help the research team to be more competitive in future proposals. Also, the research capacity developed through advancement in expertise and research infrastructure during this project will substantially increase the success rate of the research team to secure additional external research funding.
- Workshops are found to be an excellent tool for technology transfer. In the proposed study, one technology transfer workshop will be conducted.
- The findings of this study, will be published in peer-reviewed journals and conference proceedings. Although slow, such publications can have lasting impacts on technology awareness and acceptance. Publications involving the private sector is a plus.

### **Relevance to Strategic Goals**

The expected outcomes of this project are directly related to the following USDOT strategic goals: Environmental Sustainability and Economic Competitiveness. Outcomes of the proposed project will promote use of the sustainable bio-materials and agricultural byproducts as the main feedstock for production of bio-asphalt binder. This will result in a reduced need for petroleum-based binders, which leads to numerous environmental benefits. In addition, due to availability of the bio-materials and their lower cost, a significant saving in asphalt binder cost is projected as a result of this project.

## **Educational Benefits**

This project will provide a good learning opportunity for both graduate and undergraduate students. A graduate student will be working on this project as a GRA. The results of this study will be used to provide materials for his/her thesis. Undergraduate students will also be given an opportunity to work on this project. The outcomes of this study can also be used as course materials for selected lectures in the CEE 765: Pavement Design and CEE 411/511/L: Asphalt Materials and Mix Design courses.

## **Technology Transfer**

As part of implementation, it is proposed to present a workshop. Workshop will be organized in a convenient location (SDSU or SDDOT headquarters based on their availability) to allow broader participation by DOT employees, NAPA members, industry, and others. Moreover, research papers will be published and presentations will be made at the TRB Annual Meetings and other occasions for effective dissemination of findings of this study. Toward building a stronger transportation workforce, a major component of MPC mission and vision, we plan to blend research ideas and innovations in classroom.

## **Work Plan**

The proposed work plan consists of 6 major tasks as follows:

***Task 1 – Literature Review:*** Since the research on plant-based bio-materials is an evolving field, the research team will conduct a comprehensive literature review during the proposed study with a focus on plant-based bio-asphalt binders and bio-additives.

***Task 2 – Material Collection:*** The required materials for this study will be collected in close collaboration with our industry partners. Bio-fuel production waste and corn will be collected from local suppliers. Petroleum-based and plant-based bio-asphalt binders will be collected from Jebro Co. in Sioux City, IA and other material suppliers. Graded aggregates will be collected from Bowes Construction Co. in Brookings, SD.

***Task 3 – Laboratory Scale Production of Cellulose Nano-Fibers:*** Due to its mechanical strength and biocompatibility, nanocrystalline cellulose has been used as reinforcement polymer matrices for some applications. However, its use is not explored in pavement industry, mainly due to the absence of a widely accepted processing method and lack of laboratory test results. The focus of this task is to extract cellulose fibers from bio-fuel production waste and corn and improve the cellulose Nano-fiber's production method. For this purpose, alkali and enzymes will be used to retain cellulose with higher degree of polymerization, as follows: (1) oils and pigments will be removed from ground bio-fuel production waste and corn by using Soxhlet and anhydrous ethanol; (2) proteins will be removed using ethanol and 0.25% sodium sulphite; (3) starch, hemicellulose, and lignin will be removed using 5% NaOH; (4) remaining starch will be removed using enzymes, glucoamylase and xylanase and washing; and (5) finally, the solution will be used to create Nano-fibers using an in-house-built electrospinner. The above method can have yield efficiency as high as 72%.

***Task 4 – Characterization of Cellulose Nano-Fibers:*** The cellulose fiber structure, degree of polymerization, strength, and water retention will affect the final performance of the cellulose-

modified asphalt binder. For example, cellulose has been shown to improve the binding capacity of asphalt mixes and affect their dynamic properties (McDaniel, 2015). Hence, prior to mixing, the crude cellulose fiber will be characterized using facilities in BioMLab (Bio-materials and Biomechanics Lab) at SDSU, and its properties will be correlated with binder outcomes. Specifically, laser scanning microscopy (LSM) images will be used to characterize the fiber size and morphological features. The composition and degree of crystallinity will be characterized using X-Ray Fluorescence (XRF) and Raman spectroscopy. Finally, the stiffness of cellulose fiber will be obtained using Nano-indentation (NID). The results will be used to iterate and improve cellulose processing, as well as to correlate with the performance of asphalt mix, as shown in Figure 1.

***Task 5 – Characterization of Bio-Asphalt and Mixes Containing Cellulose Nano-Fiber:***

Laboratory tests will be conducted to evaluate four important performance measures of asphalt mixes produced by using plant-based bio-asphalt and cellulose-Nano fibers. These performance measures are (1) resistance to fatigue cracking; (2) resistance to rutting; (3) resistance to moisture damage; (4) stiffness; and (4) optimum plant mixing temperature. The workflow for this task is presented in Figure 2. For this purpose, a PG 58-28 asphalt binder will be mixed with 2, 5, and 7% of the cellulose Nano-fiber produced in Task 3 as an additive. Also, the collected plant-based bio-asphalt binder will be mixed with PG 58-28 asphalt binder with amounts of 0, 7, 25, 50 and 100% by the weight of binder. Then, these binder mixes will be used to design and produce 8 different types of Superpave<sup>®</sup> asphalt mixes, namely HMA-1 through 8. The produced asphalt mixes will be compacted to  $7.0\pm 0.5\%$  air voids in a Superpave<sup>®</sup> gyratory compactor (SGC). In order to characterize the resistance of asphalt mixes to fatigue cracking and rutting, semi-circular bend (SCB) and flow number (FN) tests will be conducted on HMA samples using an asphalt standard tester according to AASHTO TP 105-13 and AASHTO T 378-17 standard test methods, respectively. Hamburg wheel tracking (HWT) and tensile strength ratio (TSR) tests will also be conducted on asphalt samples to evaluate their resistance to rutting and moisture damage according to AASHTO T 324-17 and AASHTO T 283, standard test methods, respectively. Additionally, dynamic modulus (DM) test will be conducted on asphalt mixes in accordance with AASHTO T 378-17 to determine DM values as an important M-EPDG design parameter of the bio-asphalt mixes. Finally, the optimum plant mixing temperatures for asphalt binder mixes will be determined by the rotational viscometer (RV), according to AASHTO T 316-13 test method.

***Task 6. Outreach and Technology Transfer Initiatives:*** As part of implementation, it is proposed to present a workshop. Workshop will be organized in a convenient location (SDSU or SDDOT headquarters based on their availability) to allow broader participation by DOT employees, NAPA members, industry, and others. Moreover, research papers will be published and presentations will be made at the TRB Annual Meetings and other occasions for effective dissemination of findings of this study. Toward building a stronger transportation workforce, a major component of MPC mission and vision, we plan to blend research ideas and innovations in classroom.

***Task 7 – Analyze Test Results, Summarize the Findings and Report:*** After completion of the testing program, findings of this study will be compiled and analyzed. Important enhancements in the performance of the asphalt mixes containing plant-based bio-binder and cellulose Nano-

fibers will be summarized and reported as project's final report, journal publications and conference presentations.

### **Project Cost**

Total Project Costs:	\$214,875
MPC Funds Requested:	\$107,351
Matching Funds:	\$107,524
Source of Matching Funds:	SDSU and SDSU-SUN Seed Grant

### **References**

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