DEVELOPMENT OF LIGNIN-MODIFIED ASPHALT FOR USE IN CANADA

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Abstract

Climate change concerns and continuously increasing costs of bituminous pavement materials are an ongoing challenge for the pavement industry. Reduced demand for paper is an ongoing challenge for the forest industry. To help overcome these challenges, FPInnovations, with partners and collaborators involved with in the production and use of asphaltic concrete pavements, have initiated research into a lignin-modified bitumen for Canadian pavements. This greener modified bitumen shows great promise but mainly in European trials with climatic conditions and geological contexts that are different from North America. Although the Canadian study aims at analyzing the technical, economic, and environmental feasibility of lignin-modified bitumen, this paper will emphasize research on the thermo-mechanical properties of lignin-modified bitumen to optimize the bitumen mixture for the Canadian context.

Lab testing, conducted at *École de Technologie Supérieure* (ETS) laboratories, consisted, first, in evaluating the optimum lignin-bitumen ratio, mixing time and temperature, and mixing apparatus. First phases of testing were conducted on one source of lignin and both PG58-28 and PG 52-34 bitumen. Up to 50% of the bitumen was replaced by lignin. To evaluate short-term and long-term impacts of the mixtures, testing included storage stability, viscosity, measurement of rheological properties at high and low temperatures, etc. Preliminary results showed very good homogeneity of the lignin-bitumen mixture using a conventional mixer at 140°C mixing temperature, and promising thermal and mechanical properties of the optimal mix. A hot mix asphalt was formulated using the optimum lignin-modified bitumen and tested. Testing on HMA was focused on rutting, thermal cracking, compaction, resistance to moisture induced damage, etc. The new product has the potential to reduce the environmental impact of asphaltic concrete while offering enhanced performance and reduced cost.

Introduction

Climate change concerns and uncertainty of bitumen supply are an ongoing challenge for the pavement industry. Reduced demand for paper is an ongoing challenge for the forest industry. To help overcome these challenges, FPInnovations, with partners and collaborators involved with the production and use of asphalt pavements, have initiated research into a lignin-modified asphalt pavement for use in Canada.

The substitution of lignin for bitumen in binder mixtures is predicated on it being cost effective to do so and on it creating a pavement that performs as well, if not better, than unmodified bitumen. The environmental benefits from using a bioproduct in the pavement (GHG reduction, replacing an unsustainable fossil-fuel derived material with a renewable sustainably produced material) are of secondary importance to road owners and asphalt producers. However, if a greener product with comparable cost and performance can be created, it is likely to be widely adopted.

Economic potential

In pulp and paper mills, lignin, which can account for up to 25% of the mass of wood, is dissolved and becomes a "black liquor". It is burned to enhance certain products and to supply the plant with energy. According to Tilasto (2020), the Canadian paving industry currently utilizes about 4 million tonnes of bitumen per year. The current lignin production capacity in Canada is 10,000 tonnes (from only one mill) but there is a potential to rapidly grow to 160,000 tonnes per year (four mills have indicated interest in commercial scale lignin production). Bitumen producers buy bitumen in the winter at relatively low prices (e.g., $350 – $500/ tonne) and then sell it to asphalt producers during the summer paving season for higher prices (e.g., $500 - $650/ tonne). The price for bitumen paid by government in paving contracts can reach $900 per tonne. At a price ranging from $500 to $750 a tonne, lignin may already be a competitive alternative for the bitumen or asphalt producers; however, the price of lignin is anticipated to fall as production capacity grows. Pricing data is proprietary; however, these estimates were obtained and verified through discussion with several Canadian asphalt producers. The actual cost effectiveness of lignin substitution has yet to be determined and will be closely monitored during field demonstrations in 2021-22 and subsequent commercialization activities.

GHG reduction potential

A high-level, preliminary comparative environmental impact study was conducted by FPInnovations to evaluate the potential environmental benefits of using lignin as a replacement of a percentage of bitumen. Lignin produced from the Kraft pulping process (Kraft lignin) was used in the analysis. The high-level comparative environmental life cycle analysis (LCA), using the internationally accepted ISO 14040 LCA framework, was conducted for hot mix asphalt using virgin bitumen versus 20% Kraft lignin-modified bitumen.

The production of Kraft lignin in Canada was targeted for this analysis because data was readily available from two Kraft pulp mills: West Fraser's pulp mill in Hinton, AB which uses a LignoForce™ lignin extraction process, and a Resolute pulp mill located in St-Félicien, QC which, although lacking a lignin extraction process, feeds energy from burning lignin onto the electric grid of the province of Quebec.

The production of asphalt using virgin bitumen was estimated by Cobut and Essoua (2020) to generate about 262.60 kg of CO_2 equivalent per tonne. Based on the greenhouse gas (GHG) emissions documented for Kraft lignin manufacture in Quebec and Alberta, the production of lignin-modified asphalt was estimated to create about 255.88 kg of CO_2 equivalent per tonne. Based on the following assumptions:

- Alberta paving operations grow at 3% per year (Canadian bitumen use for pavements increased by 3%, on average, in 2013-2017 according to Tilasto 2020); AB demand for bitumen estimated to grow to 263,488 tonnes by 2030,
- market penetration of lignin asphalt grows to 20% of annual bitumen use by 2030, and
- paving in Canada uses 20% lignin-modified asphalt,

the demand for commercial lignin for Alberta pavements would be about 10,540 tonnes per year and the potential reduction in GHG emissions would be 2.6% (6,800 tonnes of CO_2 eq. per year). This estimate is based on relatively conservative assumptions of energy requirements for producing lignin and research is currently underway to reduce lignin extraction and drying energy requirements. Data from the field demonstrations also will allow verification of these estimates of the energy requirements and CO_2 production from lignin-modified asphalt paving.

Additional environmental advantages of using bio-asphalts include carbon sequestration and production sustainability. In terms of biogenic carbon, 1 kg of lignin sequesters 2.37 kg of CO_2 eq. Lignin is a bio-based polymer derived from Canadian forest biomass and this is a renewable resource managed according to sustainable forestry practices.

<u>Project objectives</u>

1. To establish a mixing method for, the rheological properties of, and the mechanical properties of lignin-modified bitumen.
2. To develop a lignin-modified asphalt mixture that meets or exceeds typical Canadian pavement specifications (e.g., for stability, compactibility, anti-aging, rutting resistance, low temperature cracking).
3. To assess the workability (blending and paving) of the lignin-modified asphalt.
4. To conduct full-scale testing of the lignin-modified asphalt pavement sections under Canadian climatic and traffic service conditions.
5. To quantify the cost and environmental advantages of this bio-product.

Background

Lignin is one of the main constituents of wood, the second largest after cellulose. It is the matrix that bonds together cellulose fibres in woody plants and trees. Known as the "natural glue of wood," it provides strength and stiffness. Lignin contains aromatic nuclei linked by alkyl chains like the aromatic and asphaltic fractions of bitumen, and lignin's characteristics give it the ability to act as an emulsifier or dispersant, making it potentially suitable for use in the formulation of bitumen. Researchers will seek to determine whether adding lignin to bitumen changes the properties of the bitumen or merely acts as a substitute for bitumen.

Several types of lignin from different biomass and processes were assessed prior to this study. In Canada, two manufacturing processes produce lignin: Kraft lignin (Lignoforce process) and H lignin hydrolysis (TMP-Bio process). Visually distinguishable by the colour of the lignin, all of the work to-date has been with Kraft lignin (Figure 1). Both forms of lignin eventually will be tested for use in asphalt, as they could be produced on a large scale across Canada and marketed at competitive prices. In Quebec alone, four

Kraft pulp mills could convert their residues and produce up to 150,000 tonnes per year. Although almost exclusively used for generating energy by pulp mills currently in Canada, numerous products are produced with commercial grade lignin including plywood glues, bioplastics, resins, carbon fibre composites, lignin-based gasoline and diesel, and dispersants in textile dyes (Valmet 2021).

Figure 1. Kraft lignin is a dark coloured material.

In the early 1980s, when much research was focused on alleviating the USA's dependence on foreign oil supplies, the FHWA commissioned a study by a large research team at the University of Washington State into the potential for lignin to be used as (1) a replacement for bitumen, (2) as a partial substitute for bitumen in asphalt mixtures, or (3) as an extender of emulsified asphalts in conjunction with rubber in cold mixtures. Many different combinations were created and the most promising were evaluated as bitumen mixes and in asphalt pavement mixtures. Additional analyses included environmental exposure, expected structural performance, and economic feasibility. While it was found that pavements could not be made with lignin alone, investigations (2) and (3) produced positive results. FHWA (1980) concluded that *mixtures with lignin-asphalt binders have similar and possibly improved engineering properties as compared to conventional paving mixtures*. They also concluded that *although not currently economically feasible, lignin asphalt pavements will soon be a viable alternative as the cost of asphalt increases*. While the classification of binders and the types of additives (e.g., polymers, RAP, other products) are different today than in 1980 these early investigations were with base binders similar to those used today. This early work illustrates there is potential for incorporating lignin in asphalt; however, it never reached its full potential.

Researchers from Wageningen Food & Biobased Research have created eight demonstration roads and bicycle pathways using a 50-50 blend of lignin and bitumen; some have been in service for five years and all are performing well (Gosselink et al. 2019). The intention is continue monitoring the roads to their normal lifetime of 15 years. According to Gosselink, the lignin-bitumen composite could be produced and implemented at lower temperatures than conventional asphalt (130°-140°C) by adding vegetable oil. This would save energy by reducing drying and heating costs and reducing greenhouse gas emissions. In addition, the fatigue resistance and the dynamic modulus of the lignin-modified asphalt met the requirements of the province of Zeeland in the Netherlands (the reader can refer to Gosselink et al. 2020 for further details). In 2020, Wageningen indicated that they intend to test 50-50 lignin-bitumen asphalt pavement on a heavily trafficked road, and, in the future, will target a 100% lignin pavement built with chemically modified lignin.

Van Vliet et al. (2016) illustrated changes in the phase angle with the addition of lignin at a substitution rate of 25%, and an impact on the viscoelastic behaviour of the bitumen. Lignin would tend to behave like polymer-modified binders (PMB) and would be a potential alternative in applications where improvements in rutting resistance are sought, for example.

A research group supported by the Federal Research Highway Institute (BAST) in Germany (Norgbey et al. 2020) analyzed the effects of lignin on a range of bitumen properties (characterization, morphology, decomposition, high and low temperature behaviour, fatigue resistance, deformation, aggregate adhesion in the presence of water, stability, dynamic modulus, etc.). The study concluded that the addition of lignin in the asphalt as a replacement to a percentage of bitumen allowed good cohesion, better oxidation and fatigue resistance, and better rutting resistance. In addition, the lignin-modified asphalt had better aggregate adhesion than a conventional asphalt. At 10% the addition of lignin had negligible influence on mixture segregation or compaction.

It is unclear how transferrable are the results from testing lignin in European asphalt pavements. While their binders behave just like Canadian bitumen it is difficult to directly compare European and North American binders (i.e., European binders are often not classified according to the PG system and each country also has its own classification system). In addition, much of Europe experiences warmer winters than Canada and so binders typically are stiffer than is appropriate for use in Canada.

Overview of four-step thermo-mechanical testing

All asphalt lab testing for this project was conducted by researchers Al-Falahat, Carret, and Carter at the Laboratoire sur les Chausée et Materiaux Bitumineux (LCMB) of École de Technologie Supérieure (ETS) in Montreal. LCMB was selected to perform this work because of its recognized expertise with pavement formulation, emulsions, and the incorporation of recycled materials in asphalt mixes.

The first phase of lab testing was conducted to assess the ability to mix dry lignin powder in the asphalt binder or directly into asphalt mixtures, the optimal lignin substitution rate, and the formulation of a lignin-modified asphalt surfacing mixture. Numerous combinations of bitumen PG grade, lignin substitution rate and temperature were evaluated. Testing was conducted using AASHTO, ASTM, or Quebec (LC) test standards and established the stability of the mixtures in terms of viscosity and moisture uptake, and their performance grade, rheological characteristics, and performance (low and high temperature, rutting, aging, and compactibility). Detailed results of testing are presented in (Carret, Al-Falahat, and Carter. 2021) and will be the subject of future technical papers also. The scope of this paper is limited to providing an introduction and general overview of the testing to-date and its implications.

The following work is also planned for 2021; however, it was not completed by the time of writing and, instead, will be the subject of future publications. In a second phase of lab testing, ETS will evaluate mixtures with PG52-34 bitumen (a bitumen with good low temperature performance that may help compensate for the addition of lignin without adding polymer), the effect of adding polymers, lignin coatings, mixtures using K type lignin and, subject to securing funding, the effects from using hydrophobic lignin. The NSERC industrial research Chair on the interaction of heavy loads/ climate/ pavements at Laval University (Chair i3C) will conduct accelerated pavement tests with lignin-modified asphalt pavements. The research will include assessing the workability (blending, laying, compacting) of the small batches of mixture used to pave the test section and quantifying their rutting susceptibility using an accelerated trafficking device. Two field demos of lignin-modified pavement also are planned for the fall: a new rural pavement in the City of Thunder Bay and a maintenance overlay on a road in the rural municipality of Sturgeon County near Edmonton. These field demonstrations will be used to assess longer term performance under actual Canadian climatic and traffic conditions; the paving process will also be assessed to identify potential issues in workability and to gather costs and temperature data for the analyses of life cycle costs and greenhouse gas production.

Bitumen types and lignin substitution rate

Testing was conducted on performance grade PG58-28 bitumen (a very common Canadian performance grade) and PG 52-34 (a less common performance grade that offers better low temperature performance that PG 58-28). Lignin substitution rates of 30%, 20%, 10%, and 0% were used for general assessments; however, some supplemental tests were carried out with 50% lignin substitution.

Lignin mixing

The kraft lignin for the test was extracted from the black liquor output of the TMP process at West Fraser Mills' pulp mill in Hinton, AB using a Ligno-Force™ lignin production unit. Starting at about 60% moisture content the lignin was dried to less than 1% moisture content. The dry powder was stored in a sealed container to prevent its adsorbing moisture from the air. The molecular size of the Kraft lignin was 149 microns (that is, 99.25% of test samples passed through a 150 micron sieve).

0%, 10%, 20%, 30%, and 50% lignin, by weight, was mixed with virgin bitumen in two ways: using a conventional bitumen mixer (IKA RW16 basic overhead stirrer) and by manually shaking the mixing container. The temperature in the mixer was held at 170° C while various combinations of mixing speed and mixing duration were tried; the temperature was held at 170° C while manual shaking the container for 60 minutes. The process of mixing lignin with heated bitumen is referred to in this paper as "wet mixing" (Figure 2). A mixing process of mixing lignin powder with heated pavement aggregate prior to adding in heated bitumen was also tried. This mixing process approximates the process of adding lignin into an asphalt plant pugmill after the aggregate heating section and is referred to in this paper as "dry mixing".

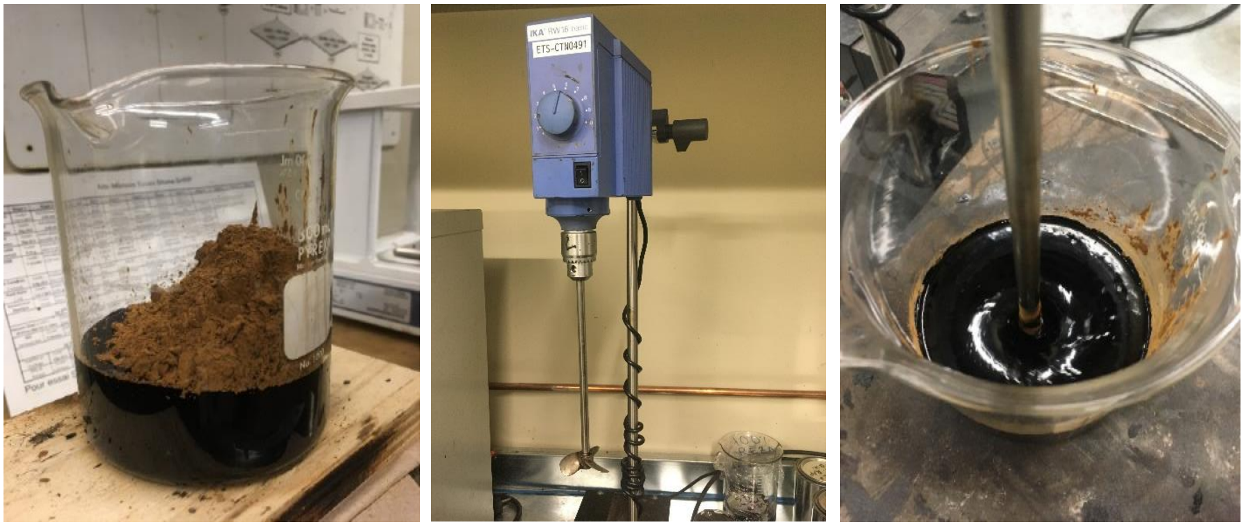

Figure 2. Mixing lignin into bitumen using a conventional asphalt mixer (Carret et al. 2021).

The dissolution of the lignin powder in the bitumen was assessed using Superpave Gyratory Compactor (SGC) air void analysis, by visual examination of the mixtures to see if they had conglomerated lignin, and by stability testing as described below. Table 1 summarizes the variables evaluated in order to determine optimal ways to create homogeneous mixing of the lignin.

Storage stability

Storage stability testing was done to assess if, and by how much, the distribution of the lignin of the lignin-bitumen mixture changes over time under the influence of environmental factors, such as temperature, humidity, and light. This also helps to quantify the homogeneity of the mixture. The test procedure (LC 25-003) consisted of conditioning tubes partly filled with bitumen at 163 °C for 48 hours and then placing them in a freezer for 3 hours. After this conditioning, thin discs of bitumen were cut at the top and at the bottom of each tube and their softening points (using AASHTO T53) were determined. The softening point is defined as the temperature at which a bitumen disc can no longer support the weight of a 3.5 g steel ball (changing from solid to liquid state). Finally, the storage stability of the bitumen corresponds to the difference between the softening point of the top disc and the bottom disc. The bitumen can be considered stable if the difference in softening temperatures is less than 3° C.

Table 1. Variables used, by test

	Lignin %, by weight	**Mixing Temperature (C)**	**Duration (minutes)**	**Mix Speeds**
SGC air void analysis	0, 10, 20, 30, 50	170°	15, 30, 60	1000 rpm
	20	170°	60	Manual shaking
Stability test	0, 10, 20, 30, 50	140°, 170°	15, 30, 60	1000 rpm
	20	170°	60	Manual shaking

For each mix presented in Table 1, two storage stability tests were performed. The stability results for the 10% and 20% lignin samples were comparable to that of the virgin bitumen and mixing temperature made little difference. The storage stability of the 10% and 20% lignin mixtures were all less than 0.8° C. In contrast, the 50% lignin formed a paste at 140° C that had low workability but was more evenly mixed at 170° C with much improved workability and storage stability of 0.4° C.

The implications of this testing were that wet mixing (adding lignin directly to bitumen) at 10% and 20% bitumen substitution rates produces satisfactory homogeneity but at substitution rates of 30% and more, increasing the mixing temperature may be needed.

Of the three mixing variables (temperature, time, and speed) mixing temperature was found to have the most impact on mixing success. Good mixability was obtained for both wet and dry mixtures of 10% lignin. Wet mixing 20% lignin resulted in very little conglomerated lignin; however, dry mixing 20% lignin was not as successful. Wet mixing 50% lignin created a paste when mixed at 140° C but at 170° C created a more uniform mix of lignin and bitumen. At this time, it was deemed premature to calculate the mixing temperature using conventional means given that the optimal lignin content has yet to be determined.

In addition to stability testing, microscope images were taken to assess lignin distribution in each sample. Microscopic images at the same scale are presented in Figure 2. It can be seen that mixing with a conventional mixer produced satisfactory dispersion of the lignin powder with very few lignin clusters in the mixture.

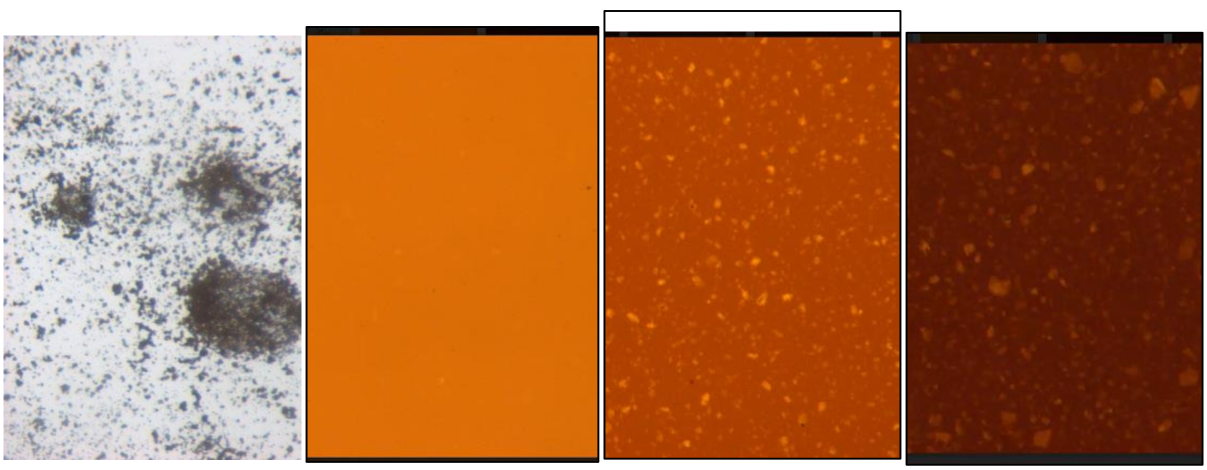

Figure 3. Microscopic pictures at the same scale (from left to right): Kraft lignin, virgin bitumen, bitumen with 10% lignin mixed at 170° C for 15 minutes, and bitumen with 20% lignin mixed at 170° C for 15 minutes.

The implications of this mix testing were that 10% to 20% dry lignin disperses well into bitumen using a conventional mixer, 170° C mixing temperature and 15 – 30 minutes of mixing. A high shear mixer is not needed. The success of manual mixing suggests that mixing lignin into a heated tanker of bitumen and using the motion of transport to blend the mixture may be feasible.[1] The success of dry mixing indicates that it is likely that the lignin could be added into the pugmill after the aggregate heating section much like any filler and this would simplify the process of adding lignin to asphalt on a commercial scale.

[1] Personal communication. Dr. Alan Carter. December 2020.

Moisture sensitivity of lignin-bitumen mixtures

The lignin-bitumen samples were tested for moisture sensitivity which was of some concern given that dried lignin adsorbs moisture from the air. For example, a sample of lignin with 1% moisture content, by weight, was left in a wet chamber for 16 days and adsorbed 13.8% water. The moisture sensitivity testing consisted of weighing samples of virgin bitumen and 50% lignin-bitumen samples left in a wet chamber to see if they gained weight the longer they were exposed to air. The results indicated that, once blended in the bitumen, the lignin was no longer able to adsorb moisture. The implications of this are that the lignin should not promote stripping in pavements and that lignin-bitumen mixtures might be suitable for product applications in which weight gain over time is not acceptable (e.g., roofing shingles); however, no stripping tests on lignin-bitumen mixtures were performed. Extraction tests are planned in follow up to determine whether the lignin was unable to adsorb moisture because it chemically bonded to the bitumen or behaves more like a filler.

Rheological properties of the lignin-bitumen mixtures

The following tests were conducted on aged and unaged lignin-modified PG 58-28 and PG 52-34 bitumen samples to determine their high and low temperature rheological properties:

- AASHTO T315 test for shear modulus and shear angle at various loads and different high and low temperatures (on unaged lignin-bitumen) using a Dynamic Shear Rheometer (DSR) machine. This test determines the key rheological properties used to determine bitumen performance grade (PG).
- AASHTO T313 flexural creep stiffness at 0° to -36° C test for low temp cracking on unaged or brittle (RAP) mixtures using a Bending Beam Rheometer (BBR) machine. The results give an indication of resistance to low temperature cracking and helps determine the PG grade at low temperature.
- AASHTO TP70 and AASHTO MP19 multiple stress creep recovery (on unaged lignin-bitumen) tests using a Dynamic Shear Rheometer (DSR) machine. Test results indicate the rutting performance of the modified bitumen.

Table 2 lists the high and low performance temperatures of PG 58-28 for various lignin substitution rates.

*Table 2. High and low temperature performance grade temperatures of the PG58-28 bitumen**

% of lignin	**PG high temp °C**	**PG low temp °C**
0	59.7	-30.9
10	60.8	-28.6
20	63.6	-26.2
30	67.3	-24.9

** Note. The virgin PG58-28 bitumen performed better than its nominal performance grade.*

The rheology of PG 52-34 bitumen was assessed also and the results are summarized in Table 3.

Table 3. High and low temperature performance grade temperatures of the PG52-34 bitumen.

% of lignin	**PG high temp °C**	**PG low temp °C**
0	52.9	-35
10	55.7	-32.7
20	58.5	-30.9

The addition of lignin to bitumen resulted in an increase in both high and low temperature performance; however, for substitution rates of less than 10% the low temperature performance is expected to be unaffected (assuming a linear increase from 0% to 10%). The increase in high and low temperature performance with the substitution of 20% lignin transformed the PG 58-28 to a PG 58-26. Interestingly, the substitution of 20% lignin transformed the PG 52-34 bitumen to a PG 58-28 – potentially creating a means to manufacture a greener form of this relatively common PG grade.

Although the low temperature performance results were undesirable, the reader will recall that these results were derived with a bitumen that does not contain polymers. Many performance grades of bitumen are normally modified with polymers or additives to improve low temperature performance (e.g., PG52-40, PG58-34, PG58-40, PG64-34, PG70-28, PG70-34) and this will be part of future testing. The focus of lignin modification testing has been on producing a low-cost product that does not need the addition of expensive polymers.

As expected, the low temperature testing for flexural creep stiffness (AASHTO T313) found that the higher the lignin content the greater the stiffness. This trend was consistent for both unaged (e.g., newly paved) modified-bitumen and aged (e.g., 7-10 years old) modified-bitumen. Detailed results can be found in (Carret et al. 2021).

The bitumen rutting tests (AASHTO TP70 and AASHTO MP19) also found that the stiffening caused by the lignin increased high temperature performance and rutting resistance. Detailed results can be found in (Carret et al. 2021). This finding agrees with Van Vliett et al. (2016) and Norgbey et al. (2020) who suggested that lignin-modified pavements may have superior performance on heavily trafficked pavements subject to high temperatures. The result also may be of importance when considering global warming and the exposure of Canadian infrastructure to longer hotter periods in the summertime.

Asphalt mixture testing (rutting, moisture sensitivity, compaction)

The mechanical performance of virgin asphalt mixes and lignin-modified asphalt mixes were evaluated with the following tests: AASHTO TP70 and AASHTO MP19 (asphalt rutting resistance tests); AASHTO T283 (moisture sensitivity test); and LC 26003 (asphalt compaction test). Samples with 20% lignin were used for this testing because this was the highest lignin concentration that had been found to have good stability and to mix easily.

The rutting performance of the asphalt sample with 20% lignin substitution was improved compared to unmodified bitumen. MSCR results found lower values of $Jnr_{3,2}$ at higher lignin contents (indicating improved resistance to rutting). Figure 4 illustrates rutting depth results for PG 58-28 conducted at 58° C. The addition of lignin improved rutting resistance for all numbers of loading cycles and 5% lignin had very similar results to 20% lignin. These results agree with the bitumen rutting resistance results and implies that rutting of lignin-modified asphalt is not likely to be an issue.

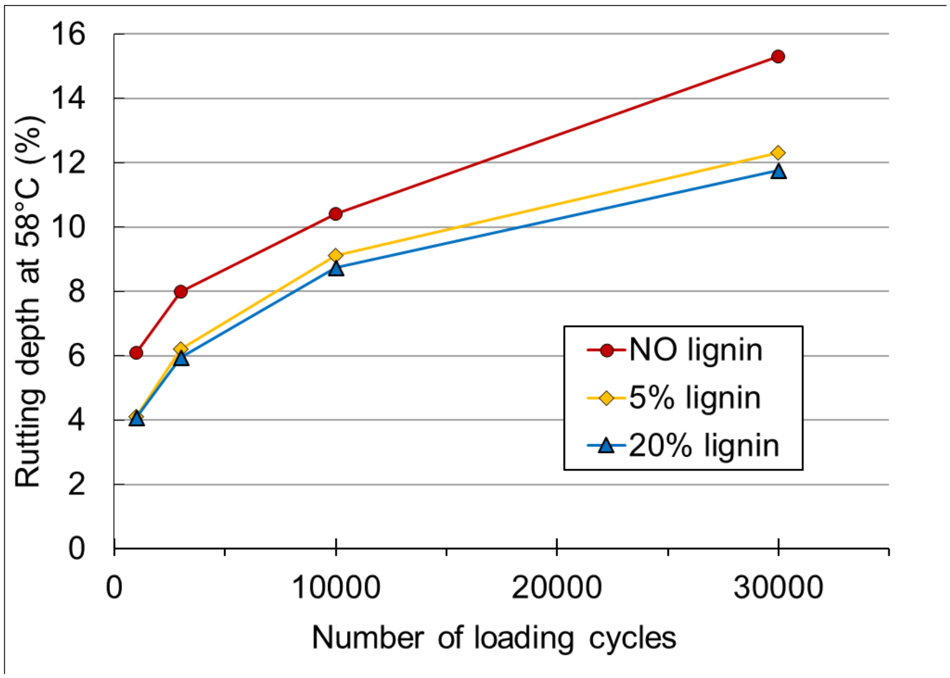

Figure 4. Rutting depth results for PG 58-28 tested at 58° C. (Carret et al. 2021).

Moisture sensitivity testing (LC 26-001) was conducted on asphalt with virgin bitumen, 5% lignin, and 20% lignin. In this test, the water resistance (WR) was determined by performing Marshall tests on dry and wet specimens. Eight Marshall specimens were compacted by applying 40 strokes on each side of the specimens with the Marshall drop hammer. The air void content of the specimens were determined and specimens were grouped in two groups of three specimens having the same average air void content. One group of specimens was tested at 25°C after a dry conditioning (about 30 minutes in a water bath at 60°C) while the other group of specimens was tested at 25°C after a wet conditioning (24 hours in a water bath at 60°C). The WR, in percent, corresponds to the ratio between the average Marshall stability of the specimens tested in wet condition with the average Marshall stability of the specimens tested in dry condition. This test gives an indication of the performance of a mix in moist conditions. Testing found that the moisture sensitivity was 82.3%, 90.8%, and 86.5% for the 0%, 5%, and 20% lignin samples. All of these exceeded the performance threshold of 70% and were, therefore, considered satisfactory. This result agrees with the bitumen moisture sensitivity results and implies that stripping of lignin-modified asphalt is not likely to be an issue.

Gyratory compaction was used to evaluate the asphaltic mixes compactibility. This test was performed on both virgin mix and both wet and dry mix lignin-modified asphalt samples with 5%, 10%, and 20% lignin. The unmodified HMA was compacted at 135° C while both wet and dry mixed samples of lignin-modified HMA were compacted at 145° C. As with the mixing temperature, it was deemed premature to calculate the compaction temperature using conventional means given that the optimal lignin content for HMA has yet to be determined. The addition of lignin increased the air void content. The wet mixed lignin-modified mix compacted to a greater extent than the dry mixed lignin-modified asphalt; however, both had higher air voids than the mix with virgin bitumen at all stages of compaction. Only the virgin asphalt and the 5%

lignin-modified mixtures met all of the requirements of LC-4202 test standard. These results indicate that compaction of lignin-modified asphalt mixes at over 5% lignin is hampered by the lignin and likely will require additional measures to reduce the air voids and achieve satisfactory pavement density (e.g., a higher compaction temperature, addition of bio-oils, higher compactive effort). Figure 6 illustrates results for the virgin PG 58-28 and 20% lignin-modified asphalt.

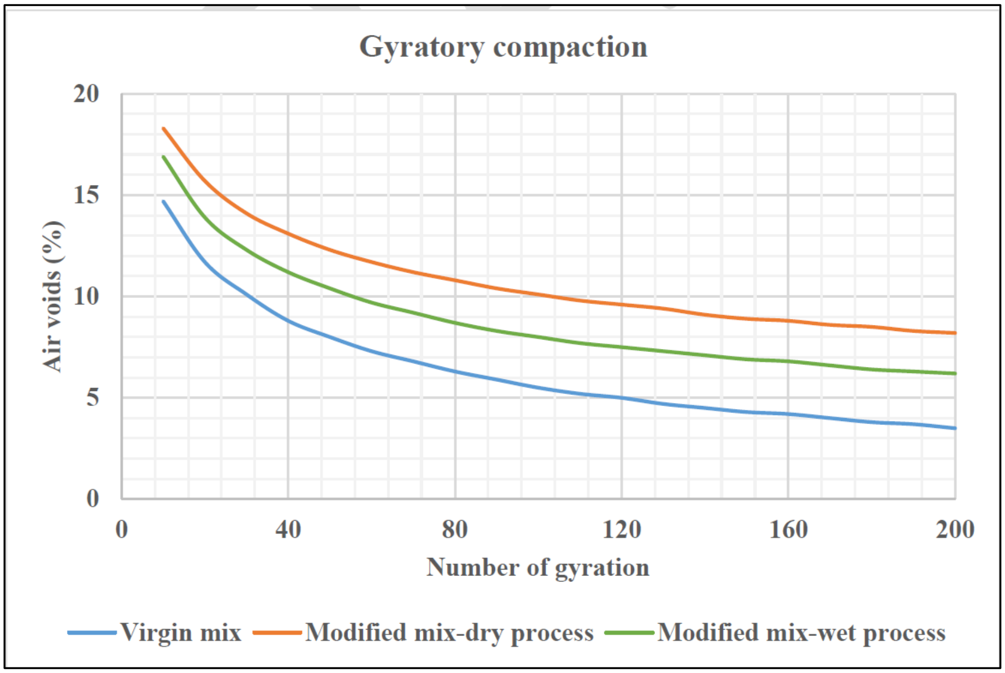

Figure 5. Gyratory compaction test results for virgin and 20% lignin-modified asphalt mixes.

Mix design for field demonstrations

LCMB researchers developed a wearing course mix design for use in the 2021 field demonstrations that features 5% lignin substitution, by weight. The relatively minor amount of lignin substitution in the asphalt is intended to generate satisfactory workability and pavement performance in these initial field trials; subsequent field trials will be conducted with higher substitution rates, assuming strategies can be developed to address performance and workability issues. Table 4 illustrates a proposed ESG wearing course design mix that incorporates 5% Kraft lignin.

Material	Mass in mix (%)
5 – 10 mm aggregates	34.1
0 – 5 mm washed aggregates	35.1
0 – 5 mm unwashed aggregates	14.2
0-1.25 mm sand	8.5
Filler	2.8
Bitumen PG 58-28	5.0
Kraft lignin	0.3

Table 4. Lignin-modified ESG mix design proposed for use in 2021 field trials.

Preliminary conclusions from thermo-mechanical testing

Lab testing of lignin-modified bitumen found the following important results:

- No special mixing apparatus is needed for mixing lignin into bitumen; however, the mixing and compaction temperatures of lignin-modified bitumen are higher. No more than 15 minutes of mixing is required to achieve homogeneous mixtures of lignin and bitumen.
- Lignin-modified bitumen and lignin-modified asphalts do not adsorb moisture from the air.
- Lignin increases the stiffness of bitumen.
- Lignin improves bitumen high temperature performance and rutting resistance.
- The lignin-modified bitumen had poorer low temperature performance than virgin PG58-28 and the low temperature performance grade temperature was decreased by 1° – 2° C for lignin substitution rates of 10% - 20%. More work needs to be done to address this performance reduction.

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