

Public Final Report

Prepared for

Alberta Innovates

Prepared by

Prof. Theo van de Ven, Sir William C. Macdonald Chair in Chemistry,
Director Quebec Centre for Advanced Materials, McGill University, Montreal QC
and Dr. Makhlouf Laleg, CEO ML Papchem Solutions Inc., Pointe-Claire QC

Project Title: Chemically Enhanced Asphaltenes as Precursors for Carbon Fibers

PROJECT INFORMATION:

Project Title:	Chemically Enhanced Asphaltenes as Precursors for Carbon Fibers
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APPLICANT INFORMATION:

Applicant (Organization):	McGill University
Address:	3420 University Street, Montreal QC H2A 3A7
Applicant Representative Name:	Theo van de Ven
Title:	Professor
Phone Number:	office: 514 398 6177, cell: 450 602 5765
Email:	theo.vandeven@mcgill.ca

PROJECT PARTNERS

Please provide an acknowledgement statement for project partners, if appropriate.

The industrial partner, ML Papchem Solutions Inc., contributed \$50K in-kind to the project

A. Executive Summary:

Provide a high-level description of the project, including the objective, key results, learnings, outcomes, and benefits

Alberta's oilsands are the third largest oil reserves in the world. These oilsands and their bitumen tend to contain a high level of heaviest non-volatile constituents, called asphaltenes, that negatively interfere with the oil refining process (high viscosity and deposition in flow lines) and make the bitumen dense. Owing to their low cost, high carbon yield, and graphene-like structure the asphaltenes are considered as potential raw materials for converting to valued carbon fibers. Alberta Innovates envisions innovation opportunities in each of the various steps required for making carbon fibers: pre-treatment, fiber spinning, stabilization, carbonization, and graphitization. This report presents relevant literature information on asphaltenes and the preliminary research results of work done under the period for Phase 1 of the CFGC project. The main project objective is to develop a cost-effective pre-treatment method that chemically enhances Alberta Oilsands Asphaltenes (AOA) to suitable asphaltene compositions for efficient spinning to uniform flexible precursor fibers in short and long discontinuous forms or continuous filaments. We have discovered three very promising approaches to make such desirable asphaltene compositions: 1) plasticized asphaltene compositions, 2) gel asphaltene compositions, and 3) aqueous asphaltene compositions. We found that with each of these explored approaches it was possible to produce asphaltene compositions capable to be converted into uniform flexible fibers of meaningful strength. The fibers produced by approach 1) or 2) can be stretched to filaments of low diameters without failure at temperatures below their melting points. The fibers made with our latent chemistries were possible to become infusible by heat treatment below their melting points. The aqueous asphaltene compositions are extremely promising, as they allow making precursor fibers at room temperature. Fibers produced with the three approaches are likely to have parallel molecular alignment / orientation in precursor fibers needed for achieving high strength carbon fibers, as well as desirable thermal and mechanical performances for adequate runnability throughout the thermo-stabilization and carbonization stages. The conditions used to develop the preliminary compositions will be further examined to show that the enhanced asphaltenes achieve tightly bonded molecules in stabilized fiber with propensity for cyclization during carbonization of fiber. Enhanced asphaltene compositions with potential success will be validated by pilot scale trials. The prospects are large that at least one of the three provisional asphaltene compositions, when further optimized and scaled-up in Phase 2 of CFGC, will become a cost-efficient technology for successfully producing carbon fibers from AOA powder with envisioned properties.

Introduction:

Please provide a narrative introducing the project using the following sub-headings.

- **Sector introduction:** Include a high-level discussion of the sector or area that the project contributes to and provide any relevant background information or context for the project.

The aim is to develop a process to produce strong carbon fibers from chemically enhanced asphaltenes or from asphaltene mixtures with a large asphaltene content. Here we summarize the relevant literature which provides insight into necessary requirements to produce such fibers.

Carbon fibers (CF), thin short or continuous filaments made predominantly from precursors polyacrylonitrile (PAN) and mesophase pitch, are well known in the industry. They are widely implemented in numerous advanced industries due to their combinations of exceptionally high tensile strength per unit weight of the fiber and superior thermal resistance and electrical properties. Most of CF entering the market is produced from PAN, with the remaining being largely derived from a pitch-type feedstock (less than 10% of the total CF in the market). The CF market is projected to grow in other important industries such as wind energy, construction, and the more traditional such as the automotive. However, rapid expansion of the application of CF in such industries will continue only if they can be produced at low cost without compromising physical properties. The carbon fibers derived from PAN, exhibit highest tensile strength but are expensive which significantly contributes the high cost of the products that include them. Carbon fibers derived from mesophase pitch show one of the highest tensile moduli of any man-made fiber, but they are brittle with short strain-to-failure, and relatively low tensile strength compared to CF derived from PAN. Mesophase pitch CF have cost and tensile strength in the range of the low strength grades of PAN-based CF. Other polymeric precursor fibers with high carbon yields are also marginally used to make low grade carbon fibers.

All CF are produced through manufacturing processes of several important stages, including selection and purification of raw material, preparation of precursor composition, spinning of composition to precursor fiber, stabilization, cyclization, carbonization of fiber and further heat treatment of graphitization, and surface sizing treatment of fiber [D.D. Edie, The effect of processing on the structure and properties of carbon fibers, *Carbon* 1998, 36, 345–362; E. Frank et al., “*Carbon fibers: Precursors, manufacturing, and properties*”. *Macromol. Mater. Eng.* 2012, 297, 493–501; M.L. Minus et al., “*The processing, properties, and structure of carbon fibers*”. *J. Miner. Met. Mater. Soc.* 2005, 57, 52–58]. The processing cost of pitch precursor (mainly pitch purification, mesophase formation, fiber spinning and stabilization) to achieve high-performance carbon fiber is higher than that of PAN precursor. The specific properties of precursors (PAN, mesophase pitch and others) and their stage processing conditions to make competitive, yet high performance carbon fibers are generally treated by the manufacturers as trade secrets.

In recent years asphaltene, the heaviest non-volatile constituents of the crude oils which have high carbon yield (78 to 90% carbon), are being explored as potential raw material to produce carbon fibers. Several literature reports, thesis and patents have been published on this subject. The chemical structure of asphaltene is difficult to ascertain due to the complex nature of asphaltene. The literature generally describes asphaltene, as non-polymeric materials, made of complex polydisperse planar compounds, not straight chains. They are thermally stable polynuclear aromatics (PNA) cores, also known as polyaromatic hydrocarbons (PAH), and contain pendant short aliphatic and alicyclic chains or groups connected to the cores by thermally unstable bonds, a small amount of polar heteroatom-containing functional groups (nitrogen, oxygen, sulfur), and heavy metals (namely organically bound vanadium, nickel). Furthermore, asphaltene compounds contain some hydroxyl and carboxyl functional groups. Asphaltene molecules (1-2 nm size) are present at different levels in all petroleum oils and their bitumen and due to their complex structure and polarity asphaltene molecules tend to self-associate to nanoaggregates (2-20 nm size) and nanoaggregate clusters (20- 120 nm size) which contain thousands of individual asphaltene molecules (K. Akbarzadeh et al., *Asphaltenes—Problematic but Rich in Potential*, *Oilfield Review* 19(2):22-43, June 2007;

M.E. Moir, 2018. *"Asphaltenes, What Art Thou? Asphaltenes and the Boduszynski Continuum"*. In the Boduszynski Continuum: *Contributions to the Understanding of the Molecular Composition of Petroleum* (pp. 3-24). American Chemical Society.) These aggregations of asphaltene constituents mainly occur when they are disrupted due to variations in chemistry, concentration, temperature, and pressure in the medium in which they are present, such as petroleum oil or bitumen, they assemble in the form of large colloidal particles that precipitate as large aggregates or flocs and deposit in flow lines (M.M. Abu-Khader et al., *"Influence of High Asphaltene Feedstocks on Processing"*, *Oil & Gas Science and Technology – Rev. IFP*, Vol. 62 (2007), No. 5). Several interactions can contribute to the formation of these clusters (E. B. Sirota et al., *"Physical Behavior of Asphaltenes"*, in *Energy & Fuels* 2007, 21, 2809-2815.) A modified Yen model study gives more information on the structural character of asphaltenes and the interfacial phenomena involving asphaltenes that might be helpful for testing future developments (O.C. Mullins, *The Modified Yen Model*, *Energy Fuels* 2010, 24, 2179–2207)

The addition of chemicals has been proposed in order to disperse and maintain in dispersion certain asphaltene constituents in crude oil or crude oil fractions in a way to dissolve them and inhibit or prohibit their aggregation / precipitation and deposition (O.H. Elochukwu, *"An environmentally friendly solvent mix for asphaltene deposit removal"*, *ARNP Journal of Engineering and Applied Sciences*, VOL. 10, NO 22, December 2015; E. Lowry et al., *"Polymers for asphaltene dispersion: Interaction mechanisms and molecular design considerations"*, *Journal of Molecular Liquids*, Volume 230, March 2017, Pages 589-599; US5,021,498; US 9,045,676 B2; WO 2016/053634 A1). In general, the structure of these chemicals consists of two main parts: polar organic backbone with functional chains or groups that favorably interact with asphaltene constituents and provide sufficient solubility in oil (hydrocarbon medium) to stabilize and prevent asphaltene precipitation.

Asphaltenes are soluble in aromatic solvents (such as toluene, xylene), but insoluble in low-boiling saturated hydrocarbons such as petroleum naphtha solvents (pentane, heptane, and hexane). Based on this solubility de-asphalting procedures are successfully used to separate asphaltenes in the form of solid particles by precipitating the asphaltene fraction from dissolved bitumen, followed by washing and drying (Lamia Goual, University of Wyoming, In book: *"Crude Oil Emulsions- Composition Stability and Characterization"*, March 2012, Chapter Petroleum Asphaltenes). Asphaltene (such as AOA powder) is currently solvent removed from bitumen, in which they are naturally suspended, to positively reduce bitumen viscosity, by an Alberta oil sands operator and it is sold at price of approximately US\$40/ton (Bitumen Beyond Combustion – Phase 2 Report, Stantec Consulting Ltd., February 12, 2018).

The solvent isolated asphaltenes, in the form of a coarse dark brown to black solid (powder) at room temperature, are friable particles having essentially no cohesiveness. The asphaltene constituents do not have a single, unique structure or specific molecular weight. They are believed to have molecular mass from about 2,000 to about 10,000, but recent published molar mass data for crude oil asphaltenes ranges from 500 to 1,000 (O. C. Mullins et al., *"Asphaltenes, heavy oils and petroleomics"*, Springer Science+Business Media, 2007.) Producing fibers from non-polymeric aromatic compounds with such range of low molecular weight would be considered impracticable. It has been suggested that spinning of an asphaltene to practical precursor fibers would not be possible if the asphaltene constituents do not entangle to form large

compounds or aggregate in some form of entangled network (A. Natarajan et al., *“Preparation of Solid and Hollow Asphaltene Fibers by Single Step Electrospinning”*, *Journal of Engineered Fibers and Fabrics*, Volume 6, Issue 2 – 2011.) Alkane side chains of asphaltene constituents, which promote asphaltene-asphaltene binding, have been suggested to be beneficial to produce precursor fibers because they increase filament cohesion and fiber strength (US 9,580,839 B2.) The bonds of pendant short aliphatic chains connected to the asphaltene cores break and form free radicals above 350°C. The loss of the pendant groups results in poor solubility and melting characteristics of the asphaltenes.

The softening point of asphaltene particles is reported to range from about 130 to 160°C, depending on the solvent separation method used and degree of purification of particles (i.e., level of residual volatile components that may act as plasticizers). The thermal behavior of four petroleum asphaltenes from different vacuum residues was studied using differential scanning calorimetry (DSC) (Y. Zhang et al., *“Observation of glass transition in asphaltenes”*, *Energy and Fuels* 2004, 18, 283-284.) It was found that asphaltenes started to melt around 150°C and attained a completely liquid state at temperatures between 220 and 240°C. Heptane-insoluble asphaltenes from different sources all showed evidence for formation of a liquid melt at temperatures between 214 and 311 °C (M.R. Gray et al., *“Melting and Fluid Behavior of Asphaltene Films at 200-500 °C”*, *Energy & Fuels* 2004, 18, 1419-1423.) Moreover, repeated DSC scans under air and oxygen showed a pronounced baseline shift in the 100-180°C temperature range, which was interpreted to correspond to standard T_g values for these asphaltenes. This thermal analysis of asphaltenes and concentration dependence on viscosity suggests that high temperatures of 220 to 240°C or plasticization might be needed to form molten blends if precursor fiber is to be made by extrusion spinning. It was reported that temperature-and concentration-dependent viscosity demonstrate that the high viscosity of asphaltene-containing systems is due to their proximity to the glass transition and the reduction of viscosity with dilution is no more than a plasticization effect, or a reduction of the glass transition with dilution (E. B. Sirota et al, *“Physical Behavior of Asphaltenes”*, *Energy & Fuels* 2007, 21, 2809-2815.)

To our knowledge solvent isolated powder asphaltenes have not been used alone as received to successfully form carbon fiber. However, an asphaltene-rich pitch has been suggested as an appropriate precursor material for carbon fibers (S.M. Aldosari et al., *Journal of Materials Research and Technology*, Volume 9, Issue 4, July–August 2020, Pages 7786-7806.) A raw pitch material can be composed of four main classes of chemical compounds: saturates (low-molecular-weight aliphatic compounds), naphthene aromatics (low-molecular-weight aromatics and saturated ring structures), polar aromatics (higher molecular weight and more heterocyclic in nature) and asphaltenes (highest molecular weight fraction in pitch with the highest aromaticity and thermally most stable.) The raw pitch must be treated under specific conditions at high temperatures to convert it to polycondensate material, which predominantly consists of domains of highly parallel, plate-like polycyclic aromatic hydrocarbons, which coalesce until 100% anisotropic material (structurally ordered optically anisotropic spherical liquid crystal called mesophase pitch (MPP). MPP is thus a complex mixture of numerous essentially aromatic hydrocarbons containing anisotropic liquid-crystalline particles (carbonaceous mesophase) detectable by optical microscopy and capable of coalescence into the bulk mesophase. Precursor mesophase pitch fibers are generally made by extrusion and melt-spinning or melt blow-spinning methods. Due to high melting temperature of mesophase pitch the corresponding and relevant process parameters are used: extrusion temperature (around 340C), and fiber spinning

temperature (around 350°C). Material parameters such as pitch viscosity, spinning speed, and temperature of post spun fiber all influence the spinning process and subsequent properties of precursor fiber and final carbon fiber. During the production of the precursor fiber important orientation and alignment of the liquid crystals of molten mesophase pitch is realized in a direction parallel to that of the fibers. Such orientation and alignment of the liquid crystals can be changed by processes such as an external flow field, i.e., the extrusion and spinning processes. The geometry and dimensions of spinneret openings not only affects fiber symmetry but also molecular orientation. The presence of this ordered structure of precursor fiber prior to stabilization and carbonization stages has been suggested as fundamental for obtaining a high-quality carbon fiber.

Additionally, the molecular alignment of liquid crystals of mesophase pitch in fibers is further fairly enhanced through the carbonization and graphitization steps. During stabilization (oxidation) stage, the precursor fibers become infusible before being exposed to the final stage high-temperature carbonization treatment (dehydrogenation, cyclisation, and cross-linking.) The oxidization temperature is slightly below the fiber softening point to keep the orientated structure and to prevent fibers from sticking to each other's. Typically, mesophase pitch fibers are heated to between 275 to 350°C for several hours to reach stabilization. Oxidation is performed in gases such as air, oxygen, oxygen-nitrogen mixture, ozone, or sulfur dioxide (Morgan P. *Carbon fiber production using a pitch-based precursor*. In: Morgan P, editor. Carbon fibers and their composites. 1st ed. Boca Raton: Taylor & Francis; 2005. p.295–324.) It was reported that the optimum oxygen content in petroleum-based pitch fibers to attain maximum tensile strength of the final carbon fiber is 8% (Zeng-Min S, Wei-Dong C, Xue-Jun Z, Wei-Pu C. *Carbon fibers from petroleum pitch*. New Carbon Mater 2005;20(1):1–7). The oxidized pitch molecules contain ketone, carbonyl, and carboxyl groups, which lead to the stronger hydrogen bonding between the adjacent molecules. The introduction of oxygen containing groups and formation of hydrogen bonding between the molecules facilitate the three-dimensional cross-linking but hinder the growth of crystallites. Furthermore, there has been no consensus previously on the purpose of the fiber stretching during the oxidation step.

US Patent 4,518,483 indicated that the use of heavy feed of aromatic residues containing two constituents (a low molecular weight oil fraction which can be distilled and a non-distillable fraction of high molecular weight asphaltene) was not desirable in contrast to mesophase pitch production. The presence of such two components in the feed, which vary significantly in their chemical, thermal, coking, and molecular weight, said to make it difficult to define process conditions suitable for the polymerization /aromatic ring condensation of the feed. To overcome this problem, the heavy feed of aromatic residues was treated to separate the largely asphaltene fraction by means of a distillation or a solvent de-asphalting. A pitch suitable for carbon artifact manufacture, such as the manufacture of carbon fibers, was then obtained by heat soaking such separated largely asphaltene fraction at a temperature of about 380-440°C for a selected period. The heat soaking was conducted in a non-oxidizing atmosphere such as a nitrogen or a hydrogen atmosphere, and all the unreacted fractions were removed to concentrate and increase the anisotropic liquid crystal fraction in the final pitch product. The produced asphaltene pitch was suggested to be useful for carbon fiber production. The purpose of the thermal treatment was to transform de-asphalted bottoms to high concentrations of asphaltene compounds into a material that has the characteristics required to be threaded and that shows good reactivity levels during the stabilization stage.

Asphaltene-rich petroleum pitches, such as commercial Ashland 240 and Ashland 260, were used for conversion into carbon fibers. For example, Ashland 240 and Ashland 260, respectively contain 64.4% and 82.7% asphaltene, 8.6% and 5.9% polar aromatic, 25.4% and 11.4% naphthalene, and 1.6% and 0% saturate. Treating Ashland 240 at 400–410°C for almost 40 h, caused approximately 50% of the pitch to be transformed into an oriented phase (mesophase pitch) mixed with the remaining 50% isotropic phase pitch [Edie DD. *Pitch and mesophase fibers*. In: Figueiredo JL, Bernardo CA, Baker RTK, Huttering KJ, editors. Carbon fibers filaments and composites. 1st ed. Alvor: Springer; 1990. p. 43–72.]. Despite the presence of 64.4% content asphaltene in initial Ashland 240, several hours and higher temperatures treatments were needed to produce pitches with increased aromaticity and higher anisotropic contents (mesophase). A second proposed method of manufacturing mesophase pitch was a solvent (quinoline and pyridine) extraction process of higher molecular weight fraction and heating in the range of 230–400°C for 10 min the insoluble portion to create a polymerized phase. This method produced high molecular weight pitch, mainly mesophase pitch, however it was suggested that the primary disadvantages of this method are the solvent cost and difficulties in ensuring 100% solvent trace removal from the mesophase pitch [US4005183].

The role of thermal treatment of asphaltene-rich feedstock for making carbon fiber is also proposed in US Patent Application #20140175688A1 (2014). In this application the asphaltene is obtained from heavy oil feedstocks undergoing upgrading in a continuous coking reactor heated to between about 450°C to about 550°C at generally low pressure. The heavy residuum is essentially comprised of oil cuts, asphaltenes, and resin residues. It was noted that such heavy residuum may also be referred to in the art as petroleum pitch and/or mesophase pitch. However, there was no information on the method used to observe such mesophase pitch. The asphaltenes was not separated by solvent de-asphalting procedure, because such procedure was considered difficult, time-consuming, and expensive. Instead, in an initial step a liquid-phase stream is removed from a continuous coking reactor, then introduced through a filter to yield a purified asphaltene rich stream. The liquid-phase stream is preferably removed from the coking reactor when the asphaltene content is between about 40% to about 70%, based upon the total weight of the liquid-phase stream taken as 100% by weight. The purified hot asphaltene-rich stream is extruded through a spinneret to yield filaments. In another option the solid-phase stream comprising 60% to about 90% asphaltenes is cooled down to as low as room temperature then dissolved in a solvent, yielding an asphaltene-rich solution, which was then spun to filaments via a spinneret. The solution or dispersion preferably comprises from about 5% to about 60% by weight asphaltenes, based upon the total weight of the solution or dispersion taken as 100% by weight. In both cases the spun filaments were passed through an inert gas stream then winded-up on spools. The inert gas crossflow may be maintained at temperature between about 200°C to about 400°C to assist with evaporation of the solvent and solidification of the filaments. The filaments were then subjected to stabilization in an air atmosphere between 200°C to about 400°C for several hours and carbonized by heating the stabilized filaments to a temperature of between 1000°C to about 1500°C in an inert atmosphere. The produced carbon fiber said to have high tensile strength, although no examples for such strength were provided.

Based on the above literature information for a solvent de-asphalting asphaltene powder to be suitable to produce carbon fiber, it must be first thermally converted to a molten asphaltene feedstock comprising an

oriented phase of highly optically anisotropic constituents like that of a molten mesophase pitch feedstock. To achieve this demand the asphaltene fraction is thermally treated in the presence of an isotropic phase under a non-oxidizing atmosphere and proper conditions of temperature and time to convert asphaltene phase to an optically anisotropic feedstock. The melt spinning process of such feedstock is optimized to further promote anisotropy because the internal atomic structure of the fiber is formed during this stage.

Electrospinning of thin diameter fibers from an asphaltene solution in toluene (40% wt/wt) has also been reported in the literature (A. Natarajan et al., “*Preparation of Solid and Hollow Asphaltene Fibers by Single Step Electrospinning*”, Journal of Engineered Fibers and Fabrics, Volume 6, Issue 2 – 2011.) It was explained that formation of such fiber from concentrated asphaltene solution was possible because of the asphaltene aggregation tendency during electrospinning. However, no strength properties were given. Furthermore, very recently, melt electrospinning was used to make precursor fiber from molten asphaltene in absence of solvent, but to this point, no process treatment conditions, and strength results are available (Z. Li et al., Department of Chemical and Materials Engineering, University of Alberta, presented during CFGC Symposium 4-5 Nov. 2020.)

Furthermore, this literature study suggests that the residual heteroatoms (N and S) in asphaltene can be evaporated during high temperature stages stabilization/carbonization and thus might be not necessary to remove them prior to fiber spinning. However, there were no discussions regarding a potential negative impact of residual metal elements (V, Ni) in asphaltene on the strength of CF.

Finally, to our knowledge commercial production of carbon fibers from asphaltenes, such as those isolated by solvent de-asphalting from Alberta’s Oilsands bitumen (Alberta Oilsands Asphaltenes, AOA) or from other asphaltene-rich products, either by melt-spinning, melt blow-spinning, dry spinning, wet spinning, melt electrospinning, or solution electrospinning has not been reported yet.

Knowledge or Technology Gaps: Explain the knowledge or technology gap that is being addressed along with the context and scope of the technical problem.

Knowledge and technology gaps are cast in form of the following questions, for which as yet there are no satisfactory answers.

1. An AOA powder as received has a thermoplastic property, melts at temperatures (230 – 275°C) to a swollen viscous and sticky product that release smoky volatiles. The molten product starts hardening as the temperature is reduced toward room temperature where it becomes very brittle and easily break down into particles. Is this temperature-viscosity relationship alone suitable for melt spinning to acceptable precursor fiber for carbon fiber?
2. The high temperature melts of AOA are highly viscous. Are too viscous AOA melts suitable for continuous smooth spinning to defect-free fiber? It is not known if spinning such viscous materials at high temperatures will produce precursor fibers with anisotropic structures.

3. Needs high temperatures melting of AOA to be done by mixing under inert environment and using atmospheric or pressurized conditions?
4. Is creating a viscoelastic asphaltene network of nanoaggregates and clusters of nanoaggregates essential to promote anisotropy in spun precursor fibers? What is the role of aggregation and entanglement in such viscoelastic network on structural uniformity, level of molecular orientation and strength of asphaltene carbon fibers?
5. To produce precursor fiber from an asphaltene stream, is it preferable for the stream to be in molten, liquid, or solution form and needs for such an asphaltene stream to be aggregated and entangled to a certain level for efficient spinning to fiber?
6. The literature suggests that an asphaltene-rich feedstock in a form like a pitch is needed and must be thermally converted to create a highly optically anisotropic polyaromatic phase like that in a mesophase pitch (molecules well oriented in the fiber axis direction). Can an AOA powder as received be simply thermally pre-treated for a certain period to form a molten spinnable mesophase asphaltene?
7. The literature does not mention chemical pretreatments of asphaltene to improve viscosity, Mw, entanglement, and anisotropy. Can the AOA powder as received be chemically treated to promote these properties? What would be the effect of such chemical treatment on melting behavior, alignment/orientation of modified asphaltene molecules and stabilization?
8. Does AOA powder become useful to make carbon fiber only if it is thermally treated in the presence of an isotropic liquid phase, under conditions like those used to produce expensive mesophase pitch?
9. Can AOA molecular alignment/orientation in the fiber axis direction be achieved by only stretching of hot spun fiber (to a desired fiber diameter).
10. The literature reports that asphaltene feedstock (pitch like) can be melt spun to fiber at a temperature range of 215-240C, but the heat stabilization of fiber can be difficult due to melting and sticking of fiber. How can the spun precursor fiber be made strong and less sticky during this thermal treatment stage? Can infusible precursor fiber be efficiently produced by simple chemical crosslinking of asphaltene molecules and such infusibility reduces oxidation stage time or eliminates it, if anisotropy is not harmed?
11. Can simple thermal compounding of asphaltene with selected solvents, plasticizer chemicals or thermoplastic polymers be used to improve the melt spinning process at lower temperature while also increasing strength and stretchability of spun fiber? Does such blending interfere with or promote anisotropy?
12. The literature does not widely discuss nor give specific information on the role of heteroatoms (N, S) and metal elements (V, Ni) in asphaltene on the spinning process and quality of carbon fiber. During stabilization and carbonization nitrogen and sulfur are expected to be driven from the asphaltene yielding essentially a “cyclic structure” carbon fiber. How big is the benefit in terms of the strength of carbon fiber if the metal elements are removed from the initial asphaltene and is the cost of removing them justifiable for such strength gain?

B. Project description:

Please provide a narrative describing the project using the following sub-headings.

- **Knowledge or Technology Description:** Include a discussion of the project objectives. **Updates to Project Objectives:** Describe any changes that have occurred compared to the original objectives of the project.
- *Initial research objectives:* 1) To produce fusible blends of thermochemically enhanced asphaltenes that may optionally be combined with some lignin, that melt at temperatures lower than that of initial asphaltenes, i.e., the fusible blends have lower melt strength than initial asphaltenes, 2) To optimize the process, such that the fusible blends contain compatibilized asphaltene molecules that allow melt spinning to uniform solid “flexible” fibers free of internal and surface defects and also contain a latent chemistry that provides the ability for the spun fibers to become thermally infusible, and 3) To produce spun fibers which have the tenacity and tensile strength needed for runnability through their production, stretching, stabilization and carbonization stages to make carbon fibers.
- In addition to the above initial three objectives (which remained unchanged), we realized that it could be particularly useful if the AOA powder, which is presently only soluble in some toxic solvents, could become soluble in aqueous solutions and common polar solvents of acceptable VOC (Volatile Organic Compounds). Therefore, a new objective was added:
 - 4) To develop a simple chemical treatment of aqueous asphaltene dispersions to make functionalized asphaltenes that can be dissolved in alkaline water or in some VOC exempts polar solvents to produce useful asphaltene compositions for fiber spinning at room temperature using wet spinning as well as dry spinning processes.

Performance Metrics: Discuss the project specific metrics that will be used to measure the success of the project.

- Completed preliminary physical and chemical tests of AOA samples that are relevant to CFGC project
- Developed chemical approaches and methods for the treatment of asphaltenes
- Produced treated asphaltenes having target viscosities for efficient spinning to solid “flexible” precursor fibers
- Demonstrated ability of stretching continuous spun precursor fibers, under specific draws and temperatures, to thin precursor filaments without breakage
- Development of three approaches to produce precursor fibers (see Section D below)
- The spun precursor fibers produced with latent chemistry become thermally infusible

D. Methodology:

Please provide a narrative describing the methodology and facilities that were used to execute and complete the project. Use subheadings as appropriate.

- In depth study of relevant literature on physical and chemical properties of asphaltenes and the treatment steps used to convert and spin asphaltenes to precursor fibers was conducted, which provided clues in how to best make precursor fibers from asphaltenes.
- All conducted experiments under Phase 1 of CFGC were performed using AOA powder samples S1 and S2 provided by Alberta Innovates. Their analytical properties were provided and deemed useful.
- Physical and chemical examinations of the received AOA powder samples S1 and S2 were performed, such as solubility, TGA, FTIR, melting properties.

- Melt-mixing treatments of AOA samples were performed to remove residual volatiles and achieve stable melt viscosity. Stretchability evaluation of produced fibers (produced by simple drawing the molten material) was performed under various temperatures to estimate fiber resistance as diameter is reduced and to observe potential defects in fiber.
- Required chemical ingredients were identified and obtained, as well as equipment for the treatment of asphaltene.
- The following three potential approaches were explored for chemical modification of AOA to produce innovative precursor asphaltene compositions that can be spun to short or continuous precursor solid “flexible” fibers having meaningful strength and which have latent chemistries to become infusible and stronger:
 1. *Reactive melt blending*: An AOA powder sample is first purified from its volatiles for a determined period by mixing of molten asphaltene under controlled temperature range and a gas flow. The asphaltene sample was then mixed with selected functional plasticizers or functional polymers and a reactive melt blending procedure was used to upgrade asphaltene to plasticized asphaltene compositions. Molten plasticized asphaltene compositions were evaluated to verify their tendency to form flexible fibers and the ability to stretch the hot fibers to thin filaments under various draw levels.
 2. *Gel blending*: An AOA powder sample is first purified from volatiles as in approach 1) above. The de-gazed asphaltene sample was cooled down to a much lower temperature range then dissolved in a solvent or a mix of solvents. A selected polymer was dissolved in the same selected solvent or mix of solvents at a determined temperature range. The two concentrated solutions were mixed to form hot gel-like homogenous compositions using a blending procedure to produce gel asphaltene compositions. The hot compositions were evaluated to verify their tendency to form continuous flexible gel fibers and their ability to stretch to thin filaments under heat and varied draw levels, while evaporating the solvent (which can be captured and re-used).
 3. *Aqueous treatment approach*: An AOA powder sample as received was dispersed in water under gentle mixing at 40 to 90°C in the presence of selected chemical reagents. After a determined reaction period the dispersed chemically modified asphaltene particles were filtered, rinsed with water, and dried in a vacuum oven at 105°C to constant weight samples. The dry samples were then characterized by FTIR and tested for their solubility in different media including aqueous alkaline water and some polar solvents. The effect of treatment on the removal of elements S and N atoms and metal elements (V and Ni) will also be examined later by energy dispersive X-ray electron microscopy. Indications are that the chemistry used to chemically modify the asphaltenes, will also remove some of the undesirable atoms. Soluble asphaltenes were formulated at room temperature with carrier soluble polymers to form “dope” compositions useful for wet spinning or dry spinning technique to continuous or short fibers. The formed fibers were dried under various temperature environments to remove water or solvent while stretching fibers to thin filaments under various draw levels.

In the above three approaches the used selected plasticizers and polymers are all intended as additives to enable the asphaltene to be spun to flexible fibers of meaningful strength and stretchability. Stretching of the spun continuous fibers is used to help align / orient the constituents of the fiber and provides the formation of tightly bonded structure. The selected additives have the ability of cyclization and fusing with asphaltene molecules during the carbonization stage to carbon fiber. It is planned that during Phase 2 of the CFGC laboratory and pilot scale equipment will be used to optimize the above asphaltene treatment approaches to efficiently enhance the asphaltene compositions to make precursor fibers useful for performing on the stabilization and carbonation stages. Needed physical and chemical examinations of the produced compositions as well as their spinning performance and properties of spun fibers will also be completed. A plan for scale-up in a carbon fiber pilot facility is already initiated with a new project partner.

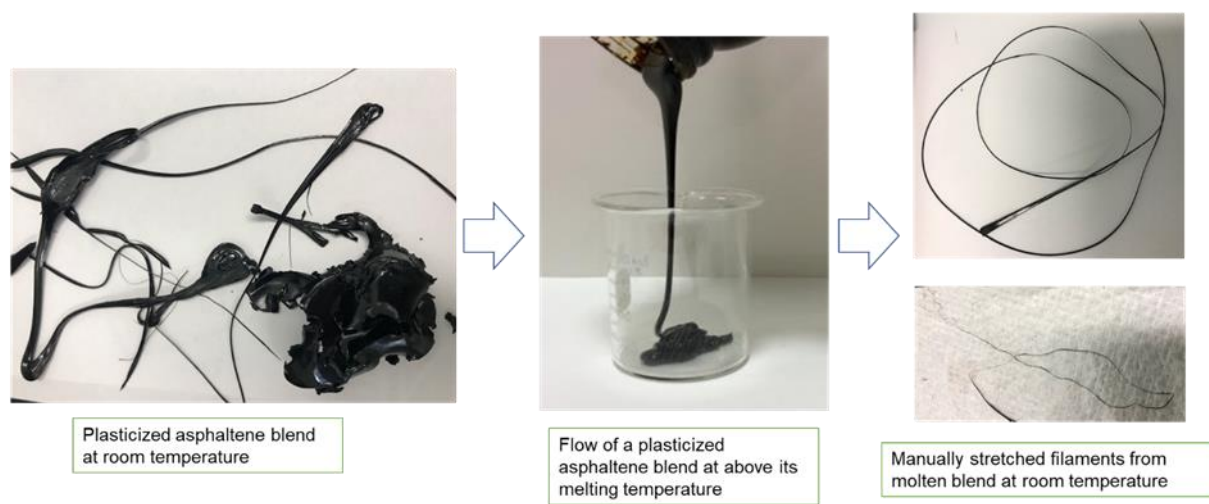
E. Project results:

Please provide a narrative describing the key results using the project's milestones as sub-headings.

- Describe the importance of the key results.
- Include a discussion of the project specific metrics and variances between expected and actual performance.
- Preliminary characterizations of AOA powder samples: Examination of the received AOA samples (S1 and S2) using FTIR, TGA under air and nitrogen, solubility in toluene and in our identified solvents and their mixtures and softening/melting characteristics indicated that they have properties like those described in the open literature. On heating they soften around 120-140°C and melt in the range of 230-270°C to a black swollen very viscous product containing voids and emits gaseous materials. The molten asphaltenes have strong tendency to stick to metal, glass, and many other solid surfaces and hardly flow on their own (without an applied high force). The viscous melt rapidly solidifies when cooled down to temperatures below softening and at room temperature it becomes extremely hard and very brittle.
- Thermal treatments of AOA samples and melt spinning to precursor fibers: Prior to melt spinning to fiber the asphaltene powder sample was treated at temperatures of 230 to 270°C for a determined period to achieve a degassed melt void-free and with a stable melt viscosity. A selected gas was injected to the mix to improve degassing and purposely enhance asphaltene physical and chemical properties. The thermally treated molten asphaltene behaved like a thermoplastic material as it was possible to draw or stretch to uniform solid fiber. The still hot fiber has some strength and was further stretched to a thin filament, but when cooled down to temperatures below its softening point it rapidly solidifies and turns to powdery particles. The thermoplastic characteristics of the thermally pre-treated asphaltene may present a narrow window of opportunity to run the hot spun precursor fiber under a temperature range around its softening point, provided that the hot spun precursor fiber has the needed strength to perform during the stabilization and carbonization stages without breaking or sticking. These requirements might be achieved by the next three experimented approaches.
- Plasticized asphaltene compositions: We identified a small number of functional aromatic oligomers and functional polymers of low Mw that, when thermally blended with an AOA powder on heating,

rapidly form a melt of reduced viscosity due to an initial plasticizing effect. Because of their compatibility with asphaltene these functional plasticizers and functional polymers cause a disruption to usually stable asphaltene aggregates causing an improvement in their mobility in the molten blend. As thermal mixing continues the reactive functional groups of plasticizers and polymers are expected to react with some available functional sites on asphaltene molecules causing them to extend. The cooled down plasticized asphaltene composition has thermoplastic characteristics and lower melting temperature and lower corresponding melt viscosity compared to the initial AOA sample. The molten plasticized asphaltene composition was easy to draw to fiber and stretch to thin filament (Fig. 1). The stretching to filament is done at a temperature around the softening point of fiber. We found that warm fiber and filament are flexible and can be banded and winded without failure. The spun fiber is purposely made with a latent chemistry and when heated under controlled temperature and environment made the fiber infusible and stronger. More work is needed to optimize the reactive melt blending of asphaltene composition and the stretching of flexible spun fiber to thin filament and verifying if such treatment promotes extended molecules and that stretching enhances alignment / orientation of such extended asphaltene in the precursor fiber. Also, it is needed to examine the rheological properties of the developed plasticized asphaltene compositions as well as to verify that polycondensation of extended asphaltene to form tightly bonded structures is achievable during stabilization of precursor fiber for efficient cyclization during carbonization.

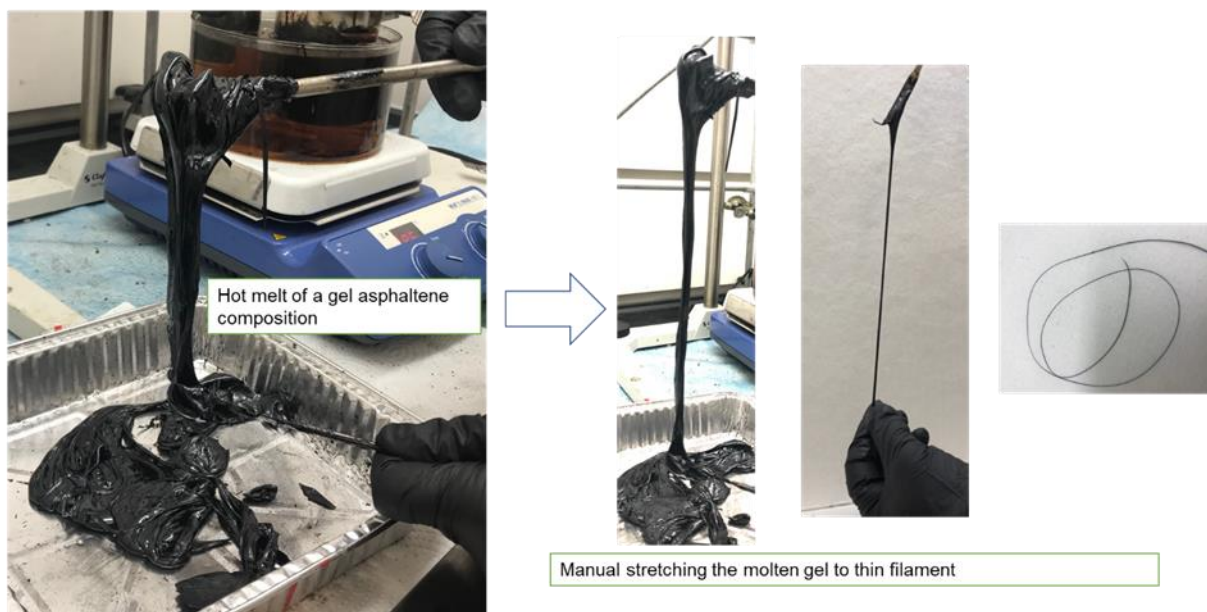
Fig. 1: Plasticized asphaltene blend and demonstration for its potential stretching to filament



Gel asphaltene compositions: We have successfully produced gel asphaltene compositions from blending dissolved AOA sample and selected dissolved polymers at controlled temperature and pressure. The identified solvents and solvent mixtures dissolved well both AOA and polymers and created uniform compositions. The formed gel compositions were easily drawn to flexible fiber under determined temperatures to form continuous flexible gel fibers of meaningful strength and stretch to break (Fig. 2). The solvent was removed from the gel fibers during their drying and stretching to solid filaments. The evaporated solvent can be recycled back to the process. During stretching the fiber diameter was reduced and a potential improvement in molecular alignment/orientation was possible. At the end of drying stage, under a determined environment, the fibers with a built-in latent chemistry

become infusible precursor solid fibers. We found that the preferred gel asphaltene compositions could only be produced by using selected high molecular weight linear polymers at specified concentration using the identified solvent that must provide compatibility amongst all components. Producing soluble asphaltene in such a gel state is an ideal method to produce satisfactory precursor fibers because the improved molecular mobility can positively enhance molecular alignment and orientation needed for carbon fiber. More work is required to examine the rheological properties of the developed gel asphaltene compositions, the degree of alignment / orientation of molecular constituents in precursor fiber caused during spinning and stretching, and the tendency to form tight bonds between asphaltene and polymer during stabilization, cyclization, and carbonization to carbon fiber.

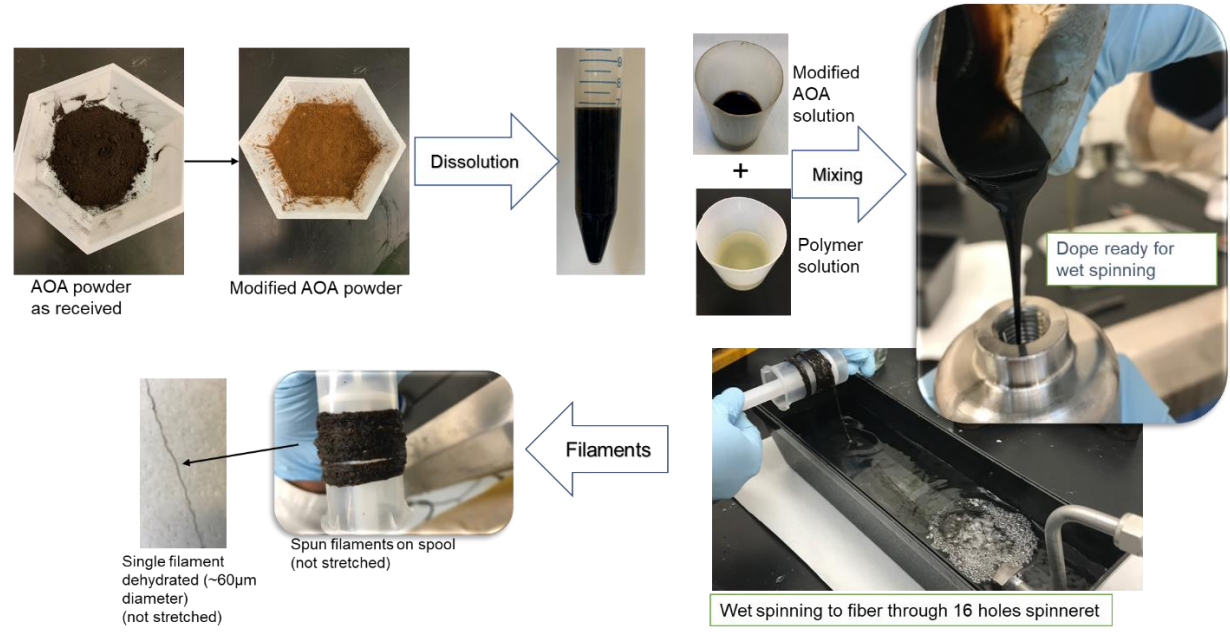
Fig. 2: Gel asphaltene composition and demonstration for its potential stretching to filament



- Aqueous asphaltene compositions:* Very recently, we explored a new cost-effective approach that uses safe chemicals under simple conditions to render AOA powder soluble in alkaline solutions and in some polar solvents at room temperature. For wet spinning applications, complete dissolution of asphaltene and all other components of a dope or a solution is needed so that they can be spun into uniform solid precursor filaments. Depending on the chemical nature and softening characteristics required, one can prepare a range of modified asphaltene compositions to meet the requirements of wet spinning to fiber and for many different other applications. We found that by using the new approach we were able to convert the AOA powder to become very soluble in alkaline solutions as well as in several polar and aprotic solvents (including acetone, methyl pyrrolidone, and several cyclic carbonates). A first modified asphaltene sample showed a 25% weight gain and its examination by FTIR show new functional groups corresponding to increased content of carboxyl and hydroxyl groups. These added groups allowed the modified asphaltene to dissolve in alkaline solution. Acidification of the alkaline solution caused the modified asphaltene to coagulate to flakes like particles. The alkaline solution containing the dissolved modified asphaltene was filtered then blended with a dissolved high molecular weight linear polymer to

produce a dope (Fig. 3). The selected polymer, which can produce precursor carbon fiber, is used here as a carrier for asphaltene molecules. The filtered dope was extruded at room temperature through a spinneret plate containing 16 holes of 100 μm diameter to an acidified water (coagulation bath) and collected on a rotating spool. The formed filaments were rinsed with ethanol and air dried under tension. The preliminary examination shows that dry filaments are flexible, have meaningful strength, and can be winded-up on a spool. The presence of new functional groups on the modified asphaltene may provide opportunities for further polymerization reactions or creating linkage between molecules for producing more enhanced asphaltene compositions for precursor fibers. Work is underway to optimize the chemical treatment process as well as thorough characterization of modified asphaltene in term of degree of functionalization and weight gain, charge titration for acid groups, molecular weight, TGA, DSC, electron microscopy (element analysis), and NMR for molecular structure.

Fig. 3: Aqueous modification of asphaltene, alkali solubility and dope preparation for wet spinning to filaments



Project Specific Metrics

Project Success Metrics (Metrics to be identified by Applicant)			
Metric	Project Target	Commercialization / Implementation Target	Comments (as needed)
Tensile strength	1500 Mpa	3000 Mpa	
Modulus	100 Gpa	300 Gpa	
asphaltene carbon fiber precursors	100 g		
Flowsheet for CF Process	1	1	Project flowsheet will differ from commercial flowsheet

The initial set objectives of the projects were all met by introducing three innovative approaches and examining them by laboratory experiments. However, due to restricted access to necessary equipment's we were not able to do all the scheduled testing of the developed modified asphaltene compositions.

F. Key Learnings:

Please provide a narrative that discusses the key learnings from the project.

- Describe the project learnings and importance of those learnings within the project scope. Use milestones as headings, if appropriate.
- The in-depth literature study suggests that for producing carbon fiber from asphaltene, such as AOA, it is essential that asphaltene is in a form like a pitch and thermally treated at specific high temperature range (320-500°C) and time, to convert it to a molten asphaltene feedstock comprising an oriented phase of optically anisotropic constituents like that of a molten mesophase pitch feedstock.
- We believe that making carbon fibers from AOA powder as received by simple melt spinning can be particularly challenging without developing solutions to the many identified hurdles.
- During this short period of the project work, done under special Covid-19 restricted conditions, we were able to develop innovative chemical treatment approaches for AOA powder and examine them in preliminary laboratory experiments that allowed us to produce asphaltene compositions potentially suitable for spinning to precursor fibers.
- We found that thermally pre-treated AOA powder as received at temperatures 230 to 290°C may present a narrow window of opportunity to make carbon fiber, provided that spun precursor fiber has some mechanical resistance to perform during the stabilization and carbonization stages without breaking or sticking. These conditions might be achieved when precursor fiber is maintained over the entire process under a temperature range around its softening point which is not practical for stabilization and carbonization stages.
- Our three chemical approaches allowed us to efficiently modify AOA powder to compositions that have the potential to be spun to flexible precursor fibers, become infusible, and have meaningful strength and stretch that are extremely promising for good runnability through stretching, stabilization, cyclization, and carbonization stages.
- Discuss the broader impacts of the learnings to the industry and beyond; this may include changes to regulations, policies, and approval and permitting processes

The plasticized and gel asphaltene compositions can be made into carbon fibers by existing commercial equipment. The chemicals used are inexpensive and widely used in other commercial applications. For the spinning of aqueous asphaltene compositions, industrial equipment is also available. The process for spinning aqueous asphaltene is similar as producing rayon fibers from a cellulose solution by extruding the solution into an acid bath. (About 5 metric ton of rayon is produced annually). The chemicals used for chemically modifying asphaltenes are equally non-expensive and widely available.

G. Outcomes and Impacts:

Please provide a narrative outlining the project's outcomes. Please use sub-headings as appropriate.

- **Project Outcomes and Impacts:** Describe how the outcomes of the project have impacted the technology or knowledge gap identified.

1. The research work of this project allowed us to overcome the critical knowledge gaps by developing innovative approaches that permitted development of three new concepts of “provisional” technologies for converting AOA powder into compositions that allow the spinning to flexible precursor fibers.
2. These “provisional” concepts, if further optimized then scaled-up, could become cost-efficient technologies for successfully producing cost-efficient & good quality carbon fibers from AOA.

The work plan consisted of three milestones, each with subtasks, goals and deliverables: 1) Producing asphaltene precursors with proven lignin process; 2) Optimization of asphaltene precursor process; and 3) Producing precursors from asphaltene and asphaltene-lignin blends. When studying the properties of asphaltenes, we realized the numerous differences between making precursor fibers from asphaltenes and lignin. As such, we concentrated more on making precursors from asphaltenes rather than from asphaltene-lignin blends, although in some compositions, lignin could be incorporated successfully. Milestones 2) and 3) were fully reached (without lignin). All the goals and deliverables were met, with the exception of the rheological properties of the compositions and the mechanical strength of the fibers. This was due to lack of access to equipment caused by the restrictions imposed to combat the Covid-19 pandemic. We had made arrangements with CO:ALIA (a research centre, specializing in plastic processing and advanced materials, associated with the CEGEP in Thetford Mines QC) during an initial visit, but the day before we were to go there to perform experiments on their equipment, a travel ban came into effect and CO:ALIA did no longer accept visitors.

- **Clean Energy Metrics:** Describe how the project outcomes impact the Clean Energy Metrics as described in the *Work Plan, Budget and Metrics* workbook. Discuss any changes or updates to these metrics and the driving forces behind the change. Include any mitigation strategies that might be needed if the changes result in negative impacts.

Clean Resources Metrics (Select the appropriate metrics from the drop down list)			
Metric	Project Target	Commercialization / Implementation Target	Comments (as needed)
\$ Future Investment			
\$ in Clean Technology	\$50,000		
# of Publications	1		after IP protection
# Students (Msc., PhD, Postdoc)	1	5	
# Patents filed	1	2	One provisional patent on lignin has been filed, a concept on asphaltenes is available and will be filed this year.
# New products/services created	1	1	asphaltene precursors for carbon fiber spinning
# New Spin-Off Companies created	0	0	

The project met the metrics above and exceeded them with a greater number of concepts for patents and expected publications.

- **Program Specific Metrics:** Describe how the project outcomes impact the Program Metrics as described in the *Work Plan, Budget and Metrics* workbook. Discuss any changes or updates to these metrics and the driving forces behind the change. Include any mitigation strategies that might be needed if the changes result in negative impacts.

Program Specific Metrics (Select the appropriate program metrics from the drop down list)			
Metric	Project Target	Commercialization / Implementation Target	Comments (as needed)
<i>Unique product/process</i>	1	1	
<i># commercial BBC products</i>	1	1	

The project developed three new approaches to preparing compositions of asphaltene feedstock for carbon fibre manufacture. These feedstocks are unique and have the potential to be commercially viable.

- **Project Outputs:** List of all obtained patents, published books, journal articles, conference presentations, student theses, etc., based on work conducted during the project. As appropriate, include attachments.

In the work plan we mentioned that the work under Phase I would lead to 1 publication, 1 additional patent (above the patent on lignin already submitted) and 1 new product. Up to now, no publications have been completed, but 3 new patents are in preparation, 2 dealing with making precursor fibers for melt and gel spinning and one dealing with the chemical modification of asphaltenes, which makes them soluble at high pH and allows extrusion in an acid bath. These 3 patents will also form the basis of 3 publications. As for new products, instead of the planned 1 new product, we have 3: thermoplastic precursors, precursors made by gel spinning and precursors made with an aqueous process. Moreover, one postdoc received training under this program.

Submission of US Provisional Applications is projected by the end of May 2021

H. Benefits:

Please provide a narrative outline the project's benefit. Please use the subheadings of Economic, Environmental, Social and Building Innovation Capacity.

- **Economic:** Describe the project's economic benefits such as job creation, sales, improved efficiencies, development of new commercial opportunities or economic sectors, attraction of new investment, and increased exports.

Producing carbon fiber from asphaltenes will lead to a new industry and jobs. Since this project is still at an early stage the economic benefits resulting from this work is too difficult to quantify.

- **Environmental:** Describe the project's contribution to reducing GHG emissions (direct or indirect) and improving environmental systems (atmospheric, terrestrial, aquatic, biotic, etc.) compared to the industry benchmark. Discuss benefits, impacts and/or trade-offs.

Making oil sands processing more efficient will have several environmental benefits, including cleaner energy production. Making efficient use of asphaltenes in novel products will be a motivating factor in separating asphaltenes from oil sands more effectively, resulting in cleaner bitumen with a reduced asphaltene content. Our developed technology is still too preliminary to know for certain how it will impact overall GHG emissions reductions.

- **Social:** Describe the project's social benefits such as augmentation of recreational value, safeguarded investments, strengthened stakeholder involvement, and entrepreneurship opportunities of value for the province.

New industrial production of carbon fibers will have social benefits, in providing job and entrepreneurship opportunities.

- **Building Innovation Capacity:** Describe the project's contribution to the training of highly qualified and skilled personnel (HQSP) in Alberta, their retention, and the attraction of HQSP from outside the province. Discuss the research infrastructure used or developed to complete the project.

The project was executed at McGill University in Montreal, Quebec. Benefits to Alberta could be that the postdoc, who has been working on the project, would certainly be willing to move to Alberta if a suitable position were to become available.

Recommendations and Next Steps

Please provide a narrative outlining the next steps and recommendations for further development of the technology developed or knowledge generated from this project. If appropriate, include a description of potential follow-up projects. Please consider the following in the narrative:

- Describe the long-term plan for commercialization of the technology developed or implementation of the knowledge generated.
- Based on the project learnings, describe the related actions to be undertaken over the next two years to continue advancing the innovation.
- Describe the potential partnerships being developed to advance the development and learnings from this project.

We have had discussions with Prof. Minoo Naebe, Institute for Frontier Materials, Carbon Nexus, Deakin University, Australia, who among other topics is working on Carbon Fibre Reinforced Composites,

Development of Low Cost Carbon Fibres and Bio-sustainable Precursor Fibres (Lignin, cellulose, etc.) and who is in charge of pilot facilities, such as a Carbon Fibre Pilot Line, a Carbonisation Process Simulator, a Precursor Spinning Line and who has unique characterization techniques, such as a Single Fibre Tensile Tester. We plan to submit a proposal for Phase II with Prof. Naebe as co-applicant. The plan is for us to optimize and fully characterize the plasticized and gel asphaltene compositions, which will be spun into first precursors fibers at the facilities at Deakin Universities and subsequently made into carbon fibers, which will be characterized by various techniques in both research groups. The aqueous compositions will be spun into fibers using the facilities present at McGill University, where our group has access to a pilot spinneret, which can run at near-industrial speeds. The spinneret is designed for textile spinning but will be upgraded to allow spinning of asphaltene-containing dopes.

At the end of Phase II, we expect to have completed all optimization and spinning conditions on a pilot facility to produce carbon fibers, which can be implemented in a full scale in Phase III for industrial production.

I. Knowledge Dissemination

Please provide a narrative outlining how the knowledge gained from the project was or will be disseminated and the impact it may have on the industry.

In our opinion, the general gained knowledge can be published in the open literature, but the three described innovative concepts that have important technological impacts should be protected through patents.

J. Conclusions

Please provide a narrative outlining the project conclusions.

- Ensure this summarizes the project objective, key components, results, learnings, outcomes, benefits, and next steps.

The main project objective was to develop a cost-effective chemical treatment method that can enhance Alberta Oil sands Asphaltenes (AOA) to produce suitable asphaltene compositions for spinning to uniform thin precursor fibers to produce short fibers and continuous filaments. The research work allowed us to overcome some of the critical knowledge gaps by developing innovative chemical approaches that permitted us to develop three new “provisional” concepts for converting AOA powder into compositions useful for spinning to precursor fibers. Each of the three identified chemical approaches was successfully examined under simple laboratory scale experiments to produce anticipated asphaltene compositions which at this point show potential properties for spinning to precursor fibers. The compositions were successfully drawn into solid yet flexible fibers using non-traditional method. The fibers could be stretched to thin filaments without failure, under specific temperatures and draw conditions. The fibers purposely contained latent chemistries that give them the needed thermal and mechanical properties for adequate runnability throughout the thermo-stabilization and carbonization stages. A latent chemistry is intended to be heat triggered prior or during stretching or stabilization stage of fiber, to form strong infusible fibers. We propose that the three initial asphaltene compositions need to be further optimized and evaluated in subsequent laboratory work for demonstrating their potential technological feasibility and satisfactory value (benefits/costs). Special focus will be on optimizing chemical reaction and blending parameters and the rheological properties of compositions, and the degree of alignment / orientation (anisotropy) of molecular constituents of compositions caused during spinning and stretching of precursor fiber.

An appropriate trial pilot scale spinning of successfully developed compositions will be performed and the produced precursor fibers examined and improved for their performance during stretching, crosslinking/stabilization and carbonization stages to deliver high strength carbon fibers of target dimensions. More work is also needed to optimize compositions to achieve tightly bonded anisotropic structures in stabilized fiber with propensity for cyclization during carbonization of fiber. Based on these preliminary findings we are confident that at least one (probably more than one) of the three provisional asphaltene compositions, if further optimized and scaled-up in Phase 2 of CFGC, could become a cost-efficient technology for successfully producing carbon fibers from AOA powder with envisioned properties. We have ensured a potential joint work with Carbon Nexus, a carbon fiber research center in Australia which has the required pilot trial facilities, for validating some of these developed concepts under Phase 2 CFGC. The concept of aqueous asphaltene compositions will be further examined and validated using McGill's wet spinning process.