

## Carbon Fibre Grand Challenge – Phase I

### Non-Confidential Project Summaries

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**Applicant** Auburn Research and Technology Foundation

**Representative** Maria Auad

**Summary** The goal of this proposal is to develop a new engineering process to produce value-added asphaltene-based carbon fibers, using Alberta Oilsands Bitumen Asphaltene (AOA) sources, with a targeted cost of less than 9 US\$/kg and a tensile strength of 3000-4000 MPa and a modulus in the range of 250-300GPa.

This proposal combines the strengths of two technological centers at Auburn University: Center for Polymers and Advanced Composites (CPAC) and the National Center for Asphalt Technology (NCAT), to develop an integrated manufacturing platform for the utilization and transformation of residual asphaltene as a base feedstock for high value-added materials.

This research will be a game-changer because i) new engineering technologies derivative from residual asphaltene will be produced, ii) carbon fibers with suitable mechanical properties at attractive cost for advanced applications will be developed, ii) a practical conversion of a low-value asphaltene waste material into high-value chemical feedstock will upgrade the value of Alberta Oilsands Bitumen for the economy while reducing the environmental impact to society.

This goal is driven by industrial opportunities, with a closely aligned focus on areas in where businesses in the composite and the asphalt industries have demonstrated an interest in the diversification of their portfolio into new product lines.

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<b>Applicant</b>	Carbon Nexus Deakin University
<b>Representative</b>	Minoo Naebe
<b>Summary</b>	<p>Carbon fibres have, over recent decades, provided for a revolution in the development of new high strength materials that are simultaneously light weight. To date, carbon fibres have mostly been manufactured from polyacrylonitrile (PAN), an expensive synthetic polymer. This has not mattered so much for high performance materials where cost is not a primary determinant; however, as the demand for new materials in more cost-sensitive applications develops pace, there is an opportunity to identify alternative raw materials.</p> <p>Alberta oil sands asphaltene (AOA) which is a combination of heavy and light polycyclic aromatics and saturated hydrocarbons with a broad molecular weight distribution is a potential carbon precursor that provide the prospect for developing a high value product from Alberta oil sands asphaltene in a market of significant size and with significant growth prospects. This project aims to develop a new class of cost effective carbon fibres using novel oilsands asphaltene derived precursors. The overarching goal of this proposal is to establish the feasibility of oilsands asphaltene as a carbon fibre feed material and develop the scientific understanding necessary to develop a low-cost production process using asphaltene derived feedstock.</p>

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<b>Applicant</b>	Chemventive LLC
<b>Representative</b>	John Newport
<b>Summary</b>	<p>The Team proposes a new method to make carbon fiber that, through a sheath core technology, will both stabilise the spinning process and prevent filament sticking downstream. The method obviates the need for a separate mesophase formation as is needed for pitch based carbon fibres.</p> <p>The entire process is amenable to computer modelling which, in conjunction with the measurement of certain Alberta Oil sands asphaltenes material properties will be used to design the required equipment and specify optimum operating conditions thus reducing development time.</p> <p>Overall this will provide a lower cost and more robust C fibre precursor spinning process.</p>

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<b>Applicant</b>	Georgia Institute of Technology
<b>Representative</b>	Satish Kumar
<b>Summary</b>	Precursor fibers will be made from Alberta asphaltene using solution or gel spinning. The project will build on the principal investigator's experience under the DARPA's Advanced Structural Fiber program for manufacturing high strength and high modulus carbon fibers. Necessary rheological additives may be incorporated to the system to achieve successful spinning, and without adding significant cost. The precursor fibers will be stabilized under appropriate time, temperature, tension, and environmental conditions followed by carbonization. Fibers will be characterized for their structure, morphology, and tensile properties. Asphaltene purity will be the key for achieving good properties for the ultimate carbon fiber.

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<b>Applicant</b>	McGill University
<b>Representative</b>	Theo van de Ven
<b>Summary</b>	Novel concept: We have developed a proprietary new concept to convert kraft lignin powder, which has no melting characteristics, to fusible blends that can be melt-spun to infusible short fibers. In particular, the infusible fibers are suitable for use as precursors to carbon fibers. We have found that chemically modified fusible lignin blends have low melt strength and thus can be melt-spun to fibers at low temperatures. Such chemical modification was achieved using a reactive melt-blending method where reactive reagents and functional thermoplastic polymers were combined with linker agents to create infusible fibers. The method of this concept was recently tested using an Asphaltene supplied by VCTek from Alberta and a compatible fusible blend was successfully produced. Here we propose to optimize the conversion of asphaltenes, that may optionally be combined with some lignin, by the same process used to make fusible lignin blends. In the proposed concept, we will investigate how various reactive plasticizers, functional thermoplastic polymers and linker agents will affect separately or combined the final properties of carbon fiber precursors. We also will vary the asphaltenes-lignin ratio (100/0 to 90/10), using a hydrogen form lignin, and the reaction conditions (speed of mixer, temperature, time). As reactive plasticizers, thermoplastic polymers and linker agents we will use those used in the production of fusible lignin, as well as related compounds.

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<b>Applicant</b>	McGill University
<b>Representative</b>	Pascal Hubert
<b>Summary</b>	Carbon fibres are used in the processing of high-performance polymer matrix composite materials. Carbon fibres are produced from carbonization of a polymeric precursor obtained from crude oil. The production of carbon fibres is controlled by companies around the world except Canada. This project aims at developing a Canadian solution to the production of carbon fibres from Alberta oilsands asphaltenes. A multidisciplinary team from McGill University, with expertise in green chemistry, soft matter characterization, polymer synthesis and composite materials processing, proposes a novel and sustainable solution to this challenge. In this proposal, minimal designer-ionic liquids will be used to dissolve the majority of asphaltene in bitumen, and then generate fibres from this mixture. Once the fibres are generated, continuous carbonization will produce carbon fibres from the ionic liquids/asphaltene mixture. This approach minimizes wastes and has the potential to significantly reduce the energy required to produce carbon fibres.

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<b>Applicant</b>	NORAM Engineering
<b>Representative</b>	James Lockhart
<b>Summary</b>	NORAM has developed a fast, low temperature, stabilization process for bitumen that will be applied to carbon fibre production from asphaltenes to reduce processing time and capital costs.

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**Applicant** National Research Council

**Representative** Chae Ho Yim

**Summary** National Research Council Canada (NRC) has years of knowledge and experience on oil sands and bitumen processing, asphaltenes products and derivatives, chemical modifications of carbon-based materials, nanomaterials productions and modifications, fibre processing using various spinning methods and carbon fibre (CF) fabrication. The NRC can thus skillfully prepare, purify, modify, transform and process Alberta oilsands asphaltenes (AOA) to enable CF production, all within one organization. Also, the NRC has the expertise and facilities to characterize and assess all materials created in every step.

AOA, as the starting material, will be engineered by initial chemical treatments followed by assembling different chemical components within AOA to produce a spinnable dope of optimized viscosity and molecular weight. Using various spinning technologies, the dope will be spun into a fibre with post-stretching treatment, and then the fibre will be subjected to cross-linking, stabilization/oxidation and carbonization processes to produce carbon fibres of various grades.

NRC will validate the initial feasibility of the developed carbon fibre process technology using an electrospinning method and its scalability to mass-production in phase 1. Alternative manufacturing methods, i.e. gel/wet/melt spinning, will be applied for scale-up in phase 2 up to the pre-commercial demonstration in phase 3.

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**Applicant** Natural Resources Canada, CanmetENERGY Devon

**Representative** Antonio De Crisci

**Summary** Sometimes described as the “cholesterol of oil”, asphaltenes are an abundant, low-value material in oil sands bitumen. Rich in carbon content and containing other elements such as nitrogen, sulfur, vanadium and nickel, the use of asphaltenes for making carbon fiber (CF) is largely under-investigated. Polyacrylonitrile (PAN) and pitch are the two main sources of CF production today, however, identifying synergies with bitumen-derived precursors for CF production could lead to lower cost, and with higher quality and properties for varied applications compared to current CF production pathways. Our proposed technology is based on utilizing bitumen-derived asphaltenes as a feedstock for CF production. The asphaltenes feedstock will be manipulated by varying the paraffinic solvent and solvent-to-bitumen ratio, creating a library of asphaltenes and determine their effect on CF quality. We will also explore the use of electrospinning and melt-electrospinning to produce high quality asphaltenes fibers for the pre-oxidation stage of CF production. Fiber size and shape control, high surface-to-volume ratio for improved thermal treating, are some of the advantages of this approach. Feedstock and product characterization, process optimization using several evaluation performance criteria such as voltage, spin rate, surface to volume ratio, solvent type, etc. and basic techno-economic studies of the proposed process will also be investigated.

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<b>Applicant</b>	Ryerson University
<b>Representative</b>	Bryan Koivisto
<b>Summary</b>	<p>Asphaltenes represent a challenge to Alberta bitumen extraction, and a tremendous opportunity as a value-added commodity in the production of new materials and technologies. Owing to their significant presence in bitumen and their propensity to aggregate, they decrease pipeline capacity and require costly flow additives. However, if they were removed at source and converted into high-value carbon fibres, this would increase pipeline capacity and serve as a secondary revenue stream for the province of Alberta. These carbon fibres could be used in a number of sustainable materials including; activated porous carbon, carbon foam, and versatile high strength composites. Each of these materials are widely used in water purification, gas adsorption, and electrochemical devices (batteries and fuel cells). This project seeks to unlock the potential of this asphaltene resource, by facilitating the low-cost conversion of asphaltenes into high-value carbon materials that can be used in the development of green technologies.</p>

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<b>Applicant</b>	University of Alberta
<b>Representative</b>	Joao Soares
<b>Summary</b>	<p>Through heat, pressure and mixing, Alberta oil sands asphaltenes may be transformed into a liquid crystalline meso-phase and then coalesce into larger anisotropic regions. The meso-phase replaces the precursor such as pitch and poly-acrylonitrile (PAN) prior to carbon fiber production. Through sudden pressure changes of the reactor during production of the meso-phase, a more efficient and environmentally friendly procedure may take place at lower temperatures. The orientation and yield of the meso-phase from asphaltenes will be observed via polarized light microscopy, scanning electron microscopy, differential scanning calorimetry and x-ray diffraction. Our strategic choice to pursue an asphaltene meso-phase allows for existing technology and infrastructure to fabricate carbon fibers to remain, lowering the capital cost and technical investment of the entire process.</p>

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**Applicant** University of Alberta

**Representative** Zhi Li

**Summary** Carbon fibers (CFs) have been widely used as the light-weight structure reinforcement materials due to their extraordinary strength and modulus. However, the mechanical properties of traditional CFs are approaching a plateau. In collaboration with our industrial partner, we propose to develop asphaltene-based ultrafine carbon fibers (UF-CFs), that bridges traditional CFs (diameter 5-7  $\mu\text{m}$ ) and carbon nanotubes (diameter 2-30 nm). The UF-CFs will be fabricated through eco-friendly melt electrospinning (e-spinning), followed by stabilization and carbonization.

Melt e-spinning has been considered as the most promising technology to produce continuous nanofibers at a large scale by avoiding the expensive solvent-removal process in conventional solution e-spinning. The tremendous elongational forces in e-spinning could significantly accelerate the molecular alignment in ABA fibers. The resulting UF-CFs are expected to possess much improved graphitic layer alignment and therefore enhanced tensile strength along the axial direction. When used as reinforcement materials, UF-CFs possess a substantial advantage over traditional CFs in matrix shear strength because their smaller cross-sectional area provides tremendous refinement and improved interaction with polymer matrices. The UF-CFs could also find applications in high-value products, such as N95 filtration materials and electrical or thermal conductive additives in energy storage and thermal management.

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**Applicant** University of Alberta

**Representative** Mohtada Sadrzadeh

**Summary** Carbon fibre (CF) is proven to be a promising material for many applications, such as aerospace, military, automobiles, and energy storage, due to its unique high tensile strength in contrast to its extremely light weight. However, the potential to capture large global market in near future is obstructed by high manufacturing cost of high-quality CF. The high manufacturing cost is imposed by high cost of raw materials, as well as tedious multistep high temperature processing. Alberta oil sand asphaltenes is a carbon rich (almost 90%) aromatic hydrocarbon can be a vital raw material in reducing the manufacturing cost of CF if the fabrication process is designed judiciously. The current project is proposed considering these two special purposes of cost and quality of asphaltene-derived CF. Floating catalyst chemical vapor deposition (FC-CVD) will be used to fabricate CF. It is a single step continuous process, utilizing a two zones heating furnace in contrast to multistep high temperature conventional methods. Vaporized carbon and catalyst precursors are injected form Zone 1, at relatively low temperature, to zone 2 where the temperature is about 800°C to grow CFs, which is collected as CNT aerogel by rotational motor rolling. Hence, adjusting the FC-CVD process for asphaltene can potentially allow fabrication of CF from this industrial waste material. CF manufacturing cost is expected to reduce substantially due to the single step low energy demanding manufacturing process.

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<b>Applicant</b>	University of Alberta
<b>Representative</b>	Cagri Ayranci
<b>Summary</b>	<p>Global sales of carbon fibre (CF) reinforced plastics are estimated to be \$48.7 billion in 2020, with the global demand for CF at 140,000 tonnes. This creates a \$4.5 billion CF market. The conventional CF precursor is polyacrylonitrile (PAN) due to its high carbon content, high molecular weight and ease of processability. However, its high cost is a disadvantage in some applications. The precursor accounts for ~53% of the CF cost driving researchers to investigate alternative precursors, such as rayon, lignin, polyethylene and pitch. Asphaltene is a strong candidate as a CF precursor due to its abundance and low cost, and high carbon content. The challenge with this material is the development of a methodology to form precursor fibres that can be converted to CF. Economically viable, i.e. with scale-up ability, CF production can be done using conventional precursor production methods; however, these are not suitable for asphaltene precursor. Our team has years of experience in small scale CF production using lignin and asphaltene. This gives us great insight, expertise, and know-how into the requirements of processing techniques, parameters and behaviour of asphaltene to form CF. We propose the use of melt-electrospinning to form asphaltene-based fibre precursors that can be converted to CF. The project has high potential to provide a value-added market for asphaltenes due to the global CF demand as well open pathways for Alberta companies to commercialize this technology.</p>

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**Applicant** University of British Columbia

**Representative** Frank Ko

**Summary** The study conducted by Alberta Innovates (AI) through the Bitumen Beyond Combustion (BBC) program concluded that carbon fibre (CF) is the most promising pathway to connect bitumen-based asphaltene, specifically Alberta oil sands asphaltenes (AOA), to value added products. However, developing the appropriate cost effective precursor and processing technologies for CF manufacturing is a major challenge for researchers and key manufacturers.

It is believed that making smaller fibres would help reduce the defects and promote quantum efficiency, surface energy and reactivity, thermal and electrical conductivity. Thus, nanofibres (NFs) have decent potential to not only address the manufacturing challenges, but also provide new opportunities for product development. Electrospinning (ES) is an excellent technique to produce the NFs. In this project, it is proposed to produce ES NFs from AOA, thermostabilize the NFs, and carbonize the NFs. The produced AOA-based CF will be studied and characterized in order to address knowledge and/or technology gaps.

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**Applicant** University of Calgary

**Representative** Jeffrey Van Humbeck

**Summary** The conversion of asphaltenes into high-performance carbon fibers is an opportunity that could deliver billions of dollars in potential revenue and provide job creation. In theory, this could be achieved by adapting the same approaches currently used to produce pitch-based fibers. However, if such an approach resulted in fibers with pitch-like properties, it may be difficult to compete with high quality fibers produced from polyacrylonitrile (PAN). We therefore propose a radically different approach.

First, beginning from full bitumen samples, we will investigate multiple separation/precipitation schemes for this complex feedstock to deliver an optimal precursor mixture. Next, we will transform the input fraction through selective oxidation. The resulting mixture—enriched in phenolic functional groups—will be co-extruded with low-cost commodity polymers (*i.e.* PET). The inclusion of minimal amounts of high molecular weight polymer can seed the microscale order necessary to yield high performance fibers and unlock true high value applications. With multiple complementary analytical techniques, we will generate data-rich descriptions of asphaltene fractions throughout the separation, oxidation, and initial fiber forming steps. Using these, we will apply current best practices in machine learning to accelerate optimization of our process steps, and to relate our characterization data to eventual carbon fiber performance.

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**Applicant** University of Calgary

**Representative** Simon Park

**Summary** Due to the volatile global oil market, there is significant needs to generate value added products from Alberta heavy hydrocarbons. This study investigates the novel methods to generate efficient carbon fibres that are comparable to pitch-based fibres. There are many unknowns and challenges associated with the production of carbon fibres (CF) from asphaltene due to its complex constitutional nature. Moreover, the conventional carbonization process requires tremendous thermal energy, thus increasing cost, time and GHG generation.

To address such issues, the proposed methods utilize new cavitation and mechanical shearing approaches to treat bitumen/asphaltene in which both methods have been proven to generate intense energy without extensive external heating. The generated energy is expected to initiate polymerization reactions to increase the quality of precursor. To further confer energy efficiency, the melt-spun fibre will be stabilized and carbonized with the combination of laser and microwave irradiations rather than the conventional oven based heating method. The laser and microwave irradiations will significantly reduce the time for the carbonization process.

Through the proposed strategy, it is expected that the reduction of the required energy for the CF production by 99%, the overall process time by 88% and reduce GHG generation by 35% relative to conventional methods. All these aspects will result in tremendous prospects for new economy in Alberta.

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**Applicant** University of Calgary

**Representative** Md Golam Kibria

**Summary** The objective of this project is to develop an economically and environmentally viable process that would enable valorization of Alberta's Asphaltenes - the organic molecules found in bitumen - into high-value carbon-fibres to create alternatives to PAN and pitch-based carbon-fibre technologies. This project has significant potential for profitable collaboration with oilsands industries in Canada and beyond. The success of the proposed project will contribute to supplying inexpensive high-strength and modulus carbon-fibre to various structural sectors, including automotive, aerospace, and construction industries. The process consists of some sub-technologies (purification, processing for spinnability, spinning optimization, and stabilization and postprocessing). Each sub-process will be optimized for the specific Alberta asphaltene to achieve the targeted technical specifications of carbon fibre. The technology in each process will be integrated in a way to minimize the energy expenditure and global carbon footprint in a cradle-to-grave model along with the cost optimization at each step. The quality of carbon fibres produced in this process will be competitive to the commercial carbon fibres in the market.

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**Applicant** University of Calgary

**Representative** Joanna Wong

**Summary** This project proposes to produce continuous carbon fibres from Alberta oilsands asphaltenes which are suitable for application in high performance structural composites. The proposed methodology is based on melt spinning, a process already used in the manufacturing of pitch fibres from mesophase pitch. Mesophase is a polymerized form of pitch which behaves as a liquid crystal and allows for high alignment of the pitch during melt spinning. In particular, this project will study the suitability of petroleum-derived asphaltenes to be melt spun, preferably without the formation of a mesophase, but also after forming a mesophase if necessary. These studies will provide information on the impact of impurities in the asphaltenes and how they affect the microstructure and degree of crystallinity of the resulting carbon fibres. Selected additives compounded into the asphaltenes will be investigated for their ability to improve the crystallinity of carbon fibres after graphitization. Maximizing the crystallinity and alignment that can be obtained from the Alberta oilsands asphaltenes-derived continuous carbon fibres is expected to result in maximum stiffness and/or strength and position these novel materials to be competitive with commercially available polyacrylonitrile and pitch-derived carbon fibres. This project comes at a highly appropriate time as it will provide a high value-added product from Alberta oilsands asphaltenes at a time when oil prices are historically low.

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**Applicant**      Zetetic Associates

**Representative**    Anthony Hladun

**Summary**      This proposal seeks to convert suitable specimens of bitumen-derived asphaltenes (provided by AI) to value-added, high performance carbon fibers (CFs). The proposing team, Zetetic Associates (Alberta, CA) and Clemson University (South Carolina, USA) has a unique combination of over 40 years of techno-economic process experience coupled with 40 years of research experience with carbon fibers derived from various precursors. Thus, a systematic approach is proposed to identify grades of low-impurity Alberta asphaltenes (low sulphur/metal impurities) that would display an optimal range of softening points (SP), i.e., low enough SP to enable melt spinning, but high enough for rapid oxidative stabilization of bitumen fibers. Optimal grades of asphaltenes will be melt spun, oxidatively stabilized, and heat treated to obtain CFs using ultrahigh temperature furnaces (2500°C). Microstructural characterization (SEM, Raman, X-ray) will determine the graphitic crystallinity, and custom-designed tensile testing equipment will be utilized to evaluate the modulus and strength of resulting high performance CFs. All of the needed equipment is fully functional within CAEFF-Clemson labs. Based on these investigations, the business case analyses of section 3.1.4 of the “Bitumen Beyond Combustion - Phase 2 Report” will be reviewed to improve the accuracy of the cost estimates for asphaltene, including cost for removal of sulphur and other contaminants.

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