ALBERTA INNOVATES

CLEAN RESOURCES FINAL REPORT PACKAGE

Project proponents are required to submit a Final Report Package, consisting of a Final Public Report and a Final Financial Report. These reports are to be provided under separate cover at the conclusion of projects for review and approval by Alberta Innovates (AI) Clean Resources Division. Proponents will use the two templates that follow to report key results and outcomes achieved during the project and financial details. The information requested in the templates should be considered the minimum necessary to meet AI reporting requirements; proponents are highly encouraged to include other information that may provide additional value, including more detailed appendices. Proponents must work with the AI Project Advisor during preparation of the Final Report Package to ensure submissions are of the highest possible quality and thus reduce the time and effort necessary to address issues that may emerge through the review and approval process.

Final Public Report

The Final Public Report shall outline what the project achieved and provide conclusions and recommendations for further research inquiry or technology development, together with an overview of the performance of the project in terms of process, output, outcomes and impact measures. The report must delineate all project knowledge and/or technology developed and must be in sufficient detail to permit readers to use or adapt the results for research and analysis purposes and to understand how conclusions were arrived at. It is incumbent upon the proponent to ensure that the Final Public Report **is free of any confidential information or intellectual property requiring protection**. The Final Public Report will be released by Alberta Innovates after the confidentiality period has expired as described in the Investment Agreement.

Final Financial Report

The Final Financial Report shall provide complete and accurate accounting of all project expenditures and contributions over the life of the project pertaining to Alberta Innovates, the proponent, and any project partners. The Final Financial Report will not be publicly released.

Alberta Innovates is governed by FOIP. This means Alberta Innovates can be compelled to disclose the information received under this Application, or other information delivered to Alberta Innovates in relation to a Project, when an access request is made by anyone in the general public.

In the event an access request is received by Alberta Innovates, exceptions to disclosure within FOIP may apply. If an exception to disclosure applies, certain information may be withheld from disclosure. Applicants are encouraged to familiarize themselves with FOIP. Information regarding FOIP can be found at http://www.servicealberta.ca/foip/. Should you have any questions about the collection of this information, you may contact the Manager, Grants Administration Services at 780-450-5551.

SEPARATION AND OXIDATION STRATEGIES FOR A NEW APPROACH TO ASPHALTENE-DERIVED CARBON FIBERS

Public Final Report

Prepared for

Alberta Innovates

Prepared by

THE UNIVERSITY OF CALGARY

JEFFREY VAN HUMBECK, ASSISTANT PROFESSOR, DEPARTMENT OF CHEMISTRY

ALBERTA INNOVATES

CLEAN RESOURCES FINAL PUBLIC REPORT TEMPLATE

1. PROJECT INFORMATION:

Project Title:	**Separation and Oxidation Strategies for a New Approach to Asphaltene-Derived Carbon Fibres**
Alberta Innovates Project Number:	G2020000349
Submission Date:	March 17, 2021
Total Project Cost:	56,229.92
Alberta Innovates Funding:	46,896.92
AI Project Advisor:	Paolo Bomben

2. APPLICANT INFORMATION:

Applicant (Organization):	**University of Calgary**
Address:	2500 University Drive NW, T2N 1N4
Applicant Representative Name:	Jeffrey Van Humbeck
Title:	Assistant Professor
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3. PROJECT PARTNERS

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

RESPOND BELOW

The University of Calgary is gratefully acknowledged for providing start-up funds to Prof. Van Humbeck that were used to purchase the GPU workstation necessary for this project.

A. EXECUTIVE SUMMARY

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

RESPOND BELOW

Our team sought to develop a representative library of well characterized asphaltene-derived materials with different chemical and physical properties from the starting whole asphaltenes provided by Alberta Innovates' sample bank. The materials we produced include fractionated asphaltenes obtained from the extraction of Alberta oil sands asphaltenes (AOA) with unusual mixtures of solvents, as well as a subset of those fractions that were further modified by catalytic oxidation. Through our solvent extractions, we were able to manipulate industrially relevant properties of the starting asphaltenes including vanadium content, sulfur content, and thermal behavior. Our selective oxidation reactions served to introduce oxygen containing functional groups to the asphaltene fractions in a precise way, modifying their molecular composition and exerting additional control over their thermal behavior. Together, the detailed collection of extracted asphaltene fractions and resulting oxidized materials may support researchers working towards carbon fiber production by expanding the range of potential asphaltene inputs, increasing the likelihood that a successful process for producing economically competitive fibers can be developed. More broadly, this library of materials may be a useful resource for exploring asphaltenes as an industrial feedstock for other novel applications.

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

RESPOND BELOW

Sector introduction

It is economically imperative for Alberta to maximize the value of its natural resources. To this end, adapting to global demand for more environmentally responsible technology by developing non-combustion applications from oil sands products is essential. Carbon fibers have been identified as an existing market that should be targeted for strategic investment – currently, the carbon fiber market is constrained by the high cost of feedstocks such as polyacrylonitrile (PAN) and expensive production processes. The conversion of AOA into a functional carbon fiber feedstock could be a commercially competitive opportunity due in large part to the asphaltenes' lower cost.

Knowledge or Technology Gaps

The conversion of AOA into high-performance carbon fibers could potentially deliver billions of dollars of economic impact. However, successful conversion of AOA into commercially viable carbon fibers faces many challenges. In theory, the conversion of asphaltenes into a functional feedstock could be achieved using the same approaches currently used to produced pitch-based fibers. However, if such an approach produced AOA-derived fibers with similar properties, it may be difficult to compete with existing high quality competitors such as fibers produced from polyacrylonitrile (PAN).

Several knowledge/technology gaps exist that prevent the immediate use of AOA in an economically competitive carbon fiber process. Unlike a pure starting material like PAN, AOA are composed of an incredible heterogeneous mixture of organic small molecules, along with inorganic impurities like nickel and vanadium. AOA sourced from different deposits in Alberta will feature different compositions, further complicating the manufacturing process. Additionally, the well-developed tools of polymer chemistry can be used in very straightforward ways to change the physical properties of PAN, adjusting the thermal and rheological behavior as desired. To the best of our knowledge, similarly advanced tools are not widely available to chemists and engineers attempting to incorporate AOA into carbon fiber production processes.

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

RESPOND BELOW

Knowledge or Technology description

We intend to support research focused on using asphaltenes as a carbon fiber feedstock by providing novel ways to transform the physical properties of the asphaltenes prior to fiber formation, through a combination of detailed separation/analysis and new chemical reaction development. To this end, the project objectives are to: (i) use the L1 and S2 asphaltenes provided by Alberta Innovates to generate a variety of asphaltene fractions through solvent extraction with multiple solvent blends; (ii) transform many of these resulting asphaltene fractions using selective oxidation reactions with scale-amenable conditions; (iii) characterize the asphaltene fractions and oxidation products using a combination of vanadium ICP-MS, IR spectroscopy, differential scanning calorimetry (DSC), sulfur elemental analysis, and high resolution mass spectrometry; (iv) create a detailed library of data which traces the impact of our solvent extractions and selective oxidations on the physical and chemical

properties of the asphaltenes, and thereby provides a comprehensive overview of potential feedstock candidates to collaborators; (v) investigate whether modern ML methods are able to relate solvent pretreatment and/or oxidation with observed properties in an actionable way. A secondary focus was placed on blending our asphaltene-derived materials with minimal amounts of inexpensive polymer to explore whether the resulting mixture was also viable as a carbon fiber precursor, as it had been brought to our attention by potential collaborators that this was an additional method of interest for carbon fiber precursor formation.

Asphaltenes are a class of compounds defined by their solubility in toluene and are composed of a complex mixture of molecules that may include polyaromatic moieties, heterocycles, alkyl chains, and trace metals such as vanadium and nickel. As such, separating and manipulating this mixture presents a clear opportunity to improve its performance as a carbon fiber precursor and increase the chances that an economically competitive process could be developed. To generate different asphaltene fractions, S2 and L1 asphaltenes would be extracted in 2-component solvent mixtures Using these novel solvent combinations, we hoped to generate a variety of interesting asphaltene fractions.

Thermal oxidative treatment is a workhorse technology in the petrochemical industry with value that cannot be overstated. However, such treatments lack the molecular precision of modern catalytic methods. We aimed here to discover whether specific combinations of solvent pretreatment and catalytic oxidation using inexpensive, earth-abundant metals could adjust the physical properties of the AOA feedstock in a way that would make it more appropriate for carbon fiber formation.

Updates to Project Objectives

The original objectives of the project were to: (i) successfully implement a separation process that can enrich a carbon fiber precursor mixture with either ‘archipelago’ or ‘island’ type asphaltene structures; (ii) develop new highly selective oxidation reactions that use inexpensive reagents and catalysts to transform the asphaltene input into a new mixture with a very precise pattern/level of oxidation; (iii) use high resolution mass spectrometry to characterize all of these mixtures in detail, so that future carbon fiber performance can be directly related to molecular structure.

Originally, the project focused on developing a selective oxidation pathway intended to convert asphaltenes into a mixture of polyphenols. Previous work in our team supported by the Canada First Research Excellence Fund (CFREF) had shown that low-cost catalysts and oxidants could deliver controlled oxidation of asphaltenes to specifically yield carbonyl products. This work was used as the starting point for developing reactions with the new L1 and S2 asphaltenes provided by AI, where we intended on extending this chemistry to include a second oxidative process to generate polyphenols. Our initial investigations with the *new* asphaltene input provided by Alberta Innovates revealed two obstacles: (i) the previously developed oxidation strategy did not generate carbonyls in these new input materials; (ii) once carbonyls were introduced to the asphaltenes using modified conditions, the solubility of the carbonyl material was poor—particularly for S1-derived fractions—which created a roadblock to some characterization methods.

We were able to successfully adjust our oxidation method to function on both S2 and L1 asphaltene inputs. Still, these findings indicated to us that controlling the nature of the asphaltene input prior to oxidation was critical. At the same time, we were able to meet many of the other participants in the Carbon Fiber Grand Challenge at the November 2020 symposium organized by AI. The initial feedback from the community indicated to us that there was a great amount of interest in our solvent extraction methods. Thus, the bulk of our data-gathering efforts shifted towards exploring scalable solvent extraction pretreatments. The intention was to creating a library of extracted materials characterized with appropriate methods that could serve as a menu of options for potential collaborators further down the carbon fiber production chain and as a starting dataset for training modern machine learning methods. A focus on asphaltene pretreatment and characterization affords the advantage of targeting different input fractions for oxidation and other applications related to optimizing asphaltenes as carbon fiber feedstocks.

Performance Metrics

The performance metrics provided in our initial proposal are restated here: Our main goal for Phase I of the Carbon Fiber Grand Challenge is to make a go/no-go decision as to whether our approach has any fatal flaws, and further, can be efficiently scaled-up to support a Phase II application with a reasonable chance to deliver on the desired performance metrics. There are four quantitative outcomes we will measure during the course of Phase I that, combined, will either support or argue against this: (i) a minimum viable quantity of oxidized materials needs to be produced (25 g); (ii) vanadium distribution during our separation processes will be quantified; (iii) sulfur amount and speciation resulting from our oxidation process will be quantified; (iv) physical/rheological properties of our phenol/polymer blends will be measured.

Based on the updates to the project objectives listed above, our metrics for success have shifted only slightly, and for the most part have been achieved. Vanadium content has been measured for 65 different asphaltene fractions that were generated through solvent extraction (including both soluble and insoluble fractions). Similarly, sulfur content was measured for an essentially identical number of prepared fractions (64). DSC measurements were performed on these extracted fractions, and approximately half of these fractions were subjected to catalytic oxidation and DSC measurements again performed after oxidation. Unmodified AOA, along with a select oxidized fraction, were blended with inexpensive commodity polymers (*e.g.* PET), and the DSC characteristics of the polymer blend were compared with that of the pure polymer.

Two potential collaborators have been identified, and upon completion of data processing, we intend to share with them the full set of collected data and offer to provide multi-gram quantities of extracted/oxidized asphaltenes that display what they consider to be desirable properties. This delivery will function to achieve performance metric (i). While not yet completed, the experience we gained to date strongly suggests that the processes will easily scale to the volume required for Phase II investigations. Our solvent extractions require only a single pass (*i.e.* not a continuous process), while our oxidation reaction does not require any specialized equipment and/or strictly controlled environmental conditions.

D. METHODOLOGY

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

RESPOND BELOW

Experimental Facilities and Characterization

Experimental work was completed in a standard chemistry lab equipped with a fumehood, heated stir plates, glassware, and chemical reagents including solvents, catalysts, and oxidizing agents. L1 and S2 asphaltene samples were provided by Alberta Innovates.

Infrared spectroscopy was completed on-site using an ATR-IR instrument accessible to the University of Calgary Chemistry department. High resolution mass spectrometry was performed in the laboratory of Prof. Stephen Larter in the Department of Geoscience at the University of Calgary. Vanadium ICP-MS was performed in the CERC laboratory for Materials Engineering for Unconventional Oil Reservoirs at the University of Calgary. Sulfur elemental analysis was provided by the Analytical and Instrumentation Laboratory at the University of Alberta. Differential scanning calorimetry was provided by the lab of Prof. Joanna Wong in the Department of Mechanical and Manufacturing Engineering at the University of Calgary.

Selective oxidation of asphaltenes

Previous work in our team had shown that low-cost catalysts and oxidants could deliver controlled oxidation of asphaltenes to specifically yield carbonyl products, as judged by FTIR analysis. To continue this work, these conditions among other reactions with precedence for selective oxidation were explored using unprocessed S2 asphaltenes as the input. Variations on reaction parameters included catalyst, pH, temperature, solvent, oxidants, and reaction time. Two robust methods were developed, and these oxidations were employed for subsequent experiments with both the S2 and L1 whole asphaltenes and solvent-extracted asphaltene fractions.

Solvent extractions

The raw L1 or S2 asphaltene was subjected to a single solvent extraction in a 2-component solvent system. 1 gram of AOA was dissolved in 30mL of solvent blend and stirred for 20-24 hours at room temperature. After this time, insoluble matter was isolated from the solution by filtration and was labelled as the ‘precipitate’ fraction. The solvent was removed from the filtrate to give the ‘soluble’ fraction. Both the soluble and precipitate fractions were analyzed by vanadium ICP-MS, sulfur elemental analysis, IR spectroscopy, and differential scanning calorimetry; the soluble fraction was additionally analyzed by high resolution mass spectrometry. Select asphaltene fractions were also subject to oxidation as per the prior outlined conditions; these fractions were analyzed by differential scanning calorimetry and IR spectroscopy.

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.

RESPOND BELOW

Milestone 1: Adapting best practices in ML process optimization

An oxidation strategy for introducing carbonyl groups to the asphaltenes was developed using the unprocessed S2 feedstock. Development began based on our team's previously established reactions which used low-cost metal catalysts and oxidants with a different asphaltene input to yield carbonyl products. These conditions were ineffective for transforming the S2 asphaltenes; upon further investigation, we discovered that increasing the pH of the reaction was key to forming carbonyls in S2, and that elevated temperatures (>50^{o}C) accelerated this process. Our final oxidation strategy still employed inexpensive catalysts and oxidant, improving the potential for its scalability. Though the reaction was developed using S2 asphaltenes as input, the strategy also exhibited good efficacy using unprocessed L1 as feedstock, as well as individual solvent-extracted fractions from both S2 and L1.

Based on the initial feedback from the asphaltene research community gained at the November 2020 symposium, we focused on producing and characterizing numerous solvent extracted materials, rather than pursuing oxidations to further transform ketone functional groups (*i.e.* to esters via Baeyer-Villiger oxidation). At the same time, our efforts in applying current machine learning approaches made a similar pivot: we are currently working on understanding/predicting solvent extraction behavior rather than further developing these oxidation processes.

In the future, with various applications perhaps demanding a variety of properties, the difference in behavior between the S2 and L1 materials provided are noteworthy. While L1 and S2 consistently demonstrated similarly significant carbonyl peaks by FTIR analysis, L1-derived materials were significantly more soluble than oxidized fractions generated from S2.

Milestone 2: Develop island/archipelago separation scheme

A total of 17 different solvent extractions were performed on the S2 asphaltenes and 18 different extractions performed on the L1 asphaltenes, giving a total of 35 extractions and 70 unique fractions. Nearly all of these fractions (64–65 of 70) were fully characterized by IR spectroscopy, differential scanning calorimetry, vanadium ICP-MS, and sulfur elemental analysis. High resolution mass spectrometry was gathered for the soluble fractions. The mass spectrometry data will serve as the basis for our attempts to adapt current machine learning approaches. Atmospheric pressure photoionization (APPi) spectra have been gather for all submitted samples and are in the process of being calibrated/quality controlled and analyzed. With other complementary characterization, we gained insight into how the chemical properties and physical behavior of the individual fractions obtained from solvent pretreatment differ from the whole asphaltene.

Vanadium: Some of our vanadium ICP-MS data is still pending. Of the data received thus far, we observed that vanadium was consistently concentrated in the precipitate fractions of both L1 and S2 asphaltenes for all solvent systems tested. For S2 asphaltenes, vanadium was reduced from 1122 ppm in

the whole asphaltene to as low as 773 ppm in soluble fractions; for L1 asphaltenes, vanadium was reduced from 231 ppm to as low as 94 ppm. This may be advantageous as depleting vanadium may improve the performance of the final carbon fiber.

Sulfur: All sulfur elemental analyses have been completed. Solvent extraction was able to deliver new fractions from S2 asphaltenes that displayed %S values ranging from 8.3 to 6.2, while those derived from L1 displayed values ranging from 8.38 to 4.28.

Some of this approach was inspired by asphaltene island/archipelago separation schemes described in the literature, which involve adsorption of the starting asphaltene onto a solid-phase adsorbent, followed by several continuous Sohxlet extractions with pure solvents of various polarity. The literature approach was tested on Cold Lake bitumen already available in-house, and the relative changes in mass spectra for our solvent-extracted fractions can be compared to these samples to determine whether simple, single-pass extraction with particular solvent blends can also enrich these structure types.

Further funding has been secured through the Canada First Research Fund to continue data processing, analysis, and the application of various ML methods to try to build an understanding of how these solvent mixtures could be used to intentionally enrich/deplete certain families of molecules. As described below in Milestone 3, even with the mass spectrometry data still being analyzed, we have already seen demonstrable impacts that different solvent extractions and oxidation methods can have on the observable properties of the asphaltene fractions.

Milestone 3: Optimize and scale-up selective oxidation

As mentioned above, in November we made the decision to focus on optimizing *pre-processing*, using two robust oxidation methods developed in Milestone 1 as our 'standard' oxidation methods for further processing/testing. Our goal was to deliver AOA fractions with appropriate physical/thermal properties that could potentially ameliorate current issues being encountered by other researchers working further down the production chain. As such, our most important goal was to observe *control* over these properties, so that we could potentially deliver whatever modification was requested. Our ability to impact these properties was initially evaluated with DSC.

Differential scanning calorimetry: For both L1 and S2, regardless of the solvent system used for extraction, both the precipitate and soluble fractions from the solvent pretreatment exhibit notably different thermal behavior from the originating whole asphaltene, and within the same solvent treatment, also differ from each other. Catalytic oxidation, further, induces significant changes to the observed behavior that we suspect other groups will be interested to investigate. Our most interesting example is provided: unmodified S2 asphaltene—in our hands—demonstrates a significant broad feature during the DSC cooling cycle between 160–140 ^{o}C. With a combination of different solvents blends, and selecting from one of our two developed oxidation conditions, this broad feature can now be replaced with a sharp one, which can be observed at 280, 260, 240 *or* 215 ^{o}C, depending on the particular solvents and catalytic reaction chosen.While we cannot yet ascribe this feature to a specific molecular feature or phase transition, the ability to use a combination of extraction and oxidation to control its position may allow the thermal/physical properties to be fine-tuned in a beneficial way.

The solvent extractions were intentionally developed as single-pass processes—rather than numerous sequential Soxhlet extractions—to simplify scalability. Similarly, our oxidation methods use

inexpensive catalysts and reagents, and no special apparatus. We have offered to produce multi-gram quantities of any AOA fraction that looks to have promising properties to 2 collaborators also working on the Grand Challenge. Successful deliver of this material in the near future will allow those groups to determine whether they want to use these modified materials in their processes currently under development, potentially in preparation for Phase II applications.

Originally, we had hoped to gather information about the oxidation state of the sulfur atoms in AOA after oxidation (*i.e.* R-S-R vs. R-SO-R vs. R-SO_2-R), where mass spectrometry would potentially be the best technique for achieving this in a quantitative way. However, we found that many of our oxidized asphaltene fractions did not have sufficient solubility for FT-ICR-MS analysis, and so this sub-goal was not pursued further.

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.
- Discuss the broader impacts of the learnings to the industry and beyond; this may include changes to regulations, policies, and approval and permitting processes

RESPOND BELOW

This project aimed to increase the fundamental understanding of simple solvent extraction and catalytic oxidation pre-treament methods on AOA. By gathering a wide range of characterization data, from two very different AOA inputs (as judged by their sample characterization forms provided), using many different extraction methods, we hoped to provide a 'menu' of carbon fiber precursor choices for engineering and materials science groups to select from. While these simple and scalable steps are not powerful enough to—for example—lower vanadium or sulfur content to trace levels, we believe that our results demonstrate that measurable changes to AOA behavior can be made.

We consider the most important observation made during our investigations to be of particular relevance. It was very surprising and noteworthy that catalytic reactions previously developed on asphaltenes prepared from Cold Lake bitumen available before project launch were entirely unsuccessful on the samples provided by Alberta Innovates. While we were able to successfully re-engineer our reaction successfully, to us this highlights the critical need for controlling the nature of the asphaltene input. We would posit that carbon fiber production trains are likely more sensitive—and more expensive to re-engineer—than our oxidation reactions. If the economic viability of a future carbon fiber production process depends on using AOA from more than one geographic location in the province, it may be the case that controlled pre-treatment is necessary, and we hope the data that we have gathered and the data analysis that is ongoing will help enable this success. Whether the changes we made to the chemical

and physical properties of AOA improve carbon fiber performance to the degree required for success is an open question for future investigations.

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.
- **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.
- **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.
- **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.

RESPOND BELOW

Project Outcomes and Impacts

Our project aimed to outline how certain features of AOA could be adjusted with simple and inexpensive pre-treatments. If it is discovered than reducing the vanadium and/or sulfur content of AOA prior to fiber production is desirable, we see few barriers to using our techniques for the next stage of the CFGC. Similarly, as better understanding is gained by other groups about how the thermal properties of the carbon fiber precursor material impact performance, starting points for modifying those properties are contained in our work.

Clean Energy Metrics

Five metrics were identified in our application's initial work plan. To the best of our knowledge, the approach we are currently pursuing to relate solvent extraction behavior with mass spectrometry information and machine learning is unique, as so we expect that the target of 1 publication will be achieved. If partners further down the production chain for carbon fiber formation identify any of our produced fractions as being ideal for scaling up, this would be an indication that a patent application is justified (application target: 1 patent). The creation of new partnerships will be pursued in the upcoming months, as we advertise our 'new service' to other teams interested in the CFGC (application targets: 1 new partnership, 1 new product/service).

Program Specific Metrics

We have identified two other academic groups that have expressed interest in investigation our pre-treated fractions further for carbon fiber production (# of end users, unique processes, and commercial BBC products targeted: 1 each).

Project Outputs

We anticipate publications/patents/presentations will develop during the upcoming year—given the short time frame of this project and the quantity of data gathered, processing and analysis are not yet completed, which need to precede these outputs.

H. BENEFITS

Please provide a narrative outline the project's benefits. 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.
- **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.
- **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.
- **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.

RESPOND BELOW

Economic and Environmental

For this project, the environmental and economic impacts are completely intertwined. As the world transitions to a low-carbon future, there can still be a place for the Alberta oilsands. It is critical to the economic health of the province that the opportunities provided by this global transition are captured. The oil sands offer a vast range of organic molecules that are currently employed predominantly for liquid fuels after upgrading, but the economic feasibility of this model is uncertain as customers begin to include carbon pricing in their equation. Transitioning these molecules into new applications that do not involve combustion will require better understanding of how to target and extract particular sub-fractions of molecules that could be well suited for a new application. This project aimed to start building that knowledge, did so efficiently with the resources and time-frame given, and has led to additional research funding being secured to further gain knowledge in the area.

Social

With the launch of the Alberta Petrochemicals Incentive Program, the provincial government has made it clear that they see opportunity in participating in more levels of the petrochemical value chain beyond the initial hydrocarbon extraction step. This work aims to provide information that will be of use to new entrants as they attempt to create valuable products from our existing natural resources.

Building Innovation Capacity

As this project transitions from data gathering to analysis and model development, the HQP hired for the project is being given the personalized instruction and opportunity to develop new skills (*i.e.* coding in python and application of modern machine learning methods) that are in high demand. The specific employment opportunity afforded by this project, and the subsequent funding it generated, has kept in Alberta a highly qualified student who was most likely going to pursue additional studies and/or employment outside the province. The provision of modern computing resources (GPU cluster time, and a dedicated lab GPU workstation) from the University of Calgary will directly support HQP training on this project and many other parallel efforts in the Van Humbeck laboratory.

I. 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.

RESPOND BELOW

This project is continuing along two parallel paths. First, two interested collaborators (one at the University of Calgary, and one at Auburn University) are going to be provided with the raw data gathered during the project. If they identify particular solvent extraction/oxidation combinations that yielded interesting sub-fractions, multi-gram quantities of those particular materials will be generated and provided for testing in actual fiber pulling experiments. With either those partners, or others that could be identified in the upcoming months, this research could potentially form part of an application for Phase II of the CFGC.

Second, we have received funding as part of the Canada First Research Excellence Fund efforts on campus to look at an expanded range of applications for AOA. Without revealing potentially sensitive information, we believe that some of the oxidized AOA fractions that we have generated could be combined with other biomaterials to deliver novel economically viable products, and we will be exploring this for at least the next year.

We intend to pursue any opportunity to share our approach and results with the local scientific community. With the launch of the Alberta Petrochemicals Incentive Program, the information that has been generated during this project may have applications of local interest that we are not yet aware of.

J. 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.

RESPOND BELOW

Once data processing has concluded, we intend to publish our approach and results, including submission to platforms that are freely accessible to the general public (e.g. ChemrXiv). Through our involvement in the campus-wide CFREF initiative, we have the opportunity to engage in many different forums with local academic and industrial partners. After patent filing (if appropriate) and publication, we will be happy to share these data as widely as possible.

K. CONCLUSIONS

Please provide a narrative outlining the project conclusions.

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

RESPOND BELOW

This project aimed to develop predictable methods for asphaltene pre-processing that could makes those materials more appropriate for carbon fiber production. The presence of undesirable impurities (*i.e.* sulfur, vanadium) and the thermal behavior—as analyzed by differential scanning calorimetry—were used to judge the degree of control our methods afforded. With a view to an economically viable process, we restricted ourselves to simple methods and equipment using inexpensive solvents, catalysts, and reagents. Empirically, these results on their own could show that a specific solvent treatment or oxidation method make AOA better suited to carbon fiber formation. A parallel goal was to try to relate the behavior that was observed with the molecular composition of the materials. We have collected high resolution mass spectrometry data for dozens of extracted fractions, and will work in the near future to determine whether an accurate predictive model that relates the solvent solvent composition used to the molecular composition of the resulting fraction can be developed with modern machine learning tools.

We have demonstrated that simple single-pass solvent extractions (*i.e.* not continuous methods) can alter sulfur and vanadium content by modest amount. In the case of the 'S2' asphaltene provided by Alberta Innovates, for example, we observed sulfur content ranging from 8.33 to 6.22 weight percent from a 7.1 weight percent starting point, according to the provided sample information. Vanadium, similarly, could be observed to range from 1768 to 773 ppm after extraction from a material containing 1122 ppm based on our internal method. Far more dramatic changes are observed in the thermal properties of these materials—particularly after oxidation. We look forward to learning from groups further down the carbon fiber production change if these modifications are beneficial.

The strength and weakness of asphaltenes as a starting material for new applications are one and the same. The incredible diversity of molecules present in these resources makes a number of commercial applications possible. Yet, the diversity of the sample—and the diversity between samples from different geographic locations—makes creating a robust process that delivers a high quality final product challenging. An increase in fundamental knowledge about Alberta oilsands asphaltenes is necessary to enable new uses, and we hope that the data gathering and analysis performed here will serve as a useful starting point for those looking to develop non-combustion products for our future economy.