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.

CLEAN RESOURCES FINAL PUBLIC REPORT TEMPLATE

1. PROJECTINFORMATION:

Project Title:	**Preparation of Feed Stock to produce Carbon Fibre from Asphaltenes**
Alberta Innovates Project Number:	G2020000390
Submission Date:	October 8, 2021
Total Project Cost:	$275,063
Alberta Innovates Funding:	$193,000
AI Project Advisor:	Paolo Bomben

2. APPLICANT INFORMATION:

Applicant (Organization):	**InnoTech Alberta**
Address:	250 Karl Clark Road, Edmonton, Alberta
Applicant Representative Name:	Pablo Contreras
Title:	Manager Hydrocarbon Processing
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Email:	Pablo.contreras@innotechalberta.ca

3. PROJECT PARTNERS

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

RESPOND BELOW

No partners

A. EXECUTIVE SUMMARY

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

RESPOND BELOW

Alberta oil producers have been facing the challenge of how to get more benefits and provide new opportunities to the industry from bitumen located in the province. Rich in asphaltenes fluids are the main group of hydrocarbons present in the bitumen that today are not utilized with efficiency. Thus, there are chances to find high-end applications to add important economic value to this waste stream and uses like carbon fibre production. The objective of this project was to develop ways to treat asphaltenes thermally and chemically to prepare feedstocks and spin the material to create filaments for future carbon fibres.

Two feedstock materials were identified from Alberta Innovates' asphaltene sample bank on the basis of preliminary properties available with Asphaltene Bank. These two feedstock materials were used as the starting base materials for subsequent feedstock preparation for carbon fibres.

The next activity was to development of suitable feedstock materials for filament spinning and for carbon fibre making process. One sample was used to obtain asphaltenes in bulk state using a solvent that precipitated asphaltenes and the other sample, with lower asphaltene content, was modified to produce feedstock materials by thermal treatment route. Autooxidation and solvent treatment steps were also explored in this project to further improve some key material characteristics. The key results from this activity include:

- Aromaticity and asphaltene content increased upon thermal treatment of vacuum bottom residue where both temperature and duration of the treatment had significant impacts.
- Product viscosity and softening points increased upon thermal treatment of vacuum bottom residue.
- High coke formation was observed at elevated thermal treatment temperatures.
- To improve material softening point and to potentially enhance aromatic and asphaltene contents, the use of a modified thermal treatment process and incorporating an autooxidation process can be done.
- Metal content reduction in the feedstock material can be achieved via solvent extraction process.

The last activity of this project was melt spinning some filaments using the modified feedstock materials. To create the carbon fibres, the spun filaments would need to be oxidized and carbonized. The oxidation and carbonization process of the filaments were not part of the scope of this project. A total of four different feedstock samples were used for melt spinning. The key results from this activity include:

- Filaments with diameter in 20-150 μm were generated from thermal treated asphaltenes samples. Some of the spun filaments were brittle or sticky depending on the feedstock material.
- Future work would explore different spinner nozzle diameters to generate fibres with consistent diameter and also investigate the impact of spinner conditions on filament properties.

The outcome of this work will facilitate the continued technology development for carbon fibre development activity which will pave way for technology development in Alberta and beyond and position Alberta at the center stage of carbon fibre feedstock development and spinning for carbon fibre development.

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

Carbon fibres have been commercially used as composite materials for several decades now. The high cost of feedstock for producing carbon fibres such as PAN hinders its applications. As a waste stream of Alberta oil production, asphaltenes have been proposed as an alternative precursor for producing low-cost carbon fibre due to their high carbon content and aromatic structure. Compared to the price of PAN at US$ 5/lb, the price of asphaltenes is only US$ 2 cents/lb, which is 250 times lower than PAN. It is expected that asphaltenes based carbon fibres will have much broader applications than the current commercial carbon fibres due to their potentially lower cost. The properties of the resultant fibres are affected by many factors such as crystallinity, molecular orientation, carbon content, impurities, etc.

There are still a few technical gaps that hinder the application of asphaltenes-rich feedstocks for carbon fibre production. In particular, feedstock sources and pre-treatment of asphaltenes-rich feedstocks impact the ability to spin asphaltenes fibre and the subsequent carbon fibre preparation significantly. Therefore, a deep characterization, additional to the basic analysis, of the feedstock in terms of chemical composition and rheological behavior is necessary. Presence of impurities in the material after the modification, such as heavy metals and sulfur, and dominant molecular structures need to be quantified and characterized. These undesirable components may potentially impact properties of spun fibres and may further lead to manufacturing issues with carbon fibers at high temperatures. Viscoelastic material properties, more than just bulk phase viscosity at process temperature as a basic analysis, rheological properties of the feedstock at different conditions will be required. This information will be key for fibre spinning process.

Feedstock chemical compositional information and rheology is key to improve feedstock quality and spinning characteristics for determination of mechanical properties and to define future industrial processes. Inappropriate feedstock preparation will likely result in poor aromatic phase development within asphaltene-based feedstock resulting in poor spinnability. Poor spinnability will lead to poor stabilization and fibre oxidation to produce carbon fibres. If the material is not properly modified and stabilized it is quite likely that the material may not be of spinning quality and cannot be turned into stable fibres. This project will address the gaps described above.

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

The objective of this project is to develop ways to treat asphaltenes thermally and chemically to prepare a feedstock that enables high performance carbon fibre production from Alberta heavy oils and bitumen sourced asphaltic material production facilities or refineries and upgraders. This is, feedstock preparation, the main technical gap to be closed for the transformation of the raw material to an appropriate material for carbon fibre. The feedstock will be spun to produce the asphaltenes-based filaments. Certain physical and chemical analysis will be completed to confirm the required properties of the feedstock for making carbon fibre.

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

An experimental approach/ methodology was adopted for this project. The activities allowed identifying appropriate feedstock starting materials and modifying them through various means to produce a range of modified feedstocks that were rich in aromatics, high asphaltenes, low metal contents and had improved softening points. Once the feedstocks were in place, a few were chosen to spin filaments. Specific conditions of sample treatment and spinning are proprietary and not disclosed in this report.

Material properties were determined at every step of material modification and a mix of in house techniques and standard methods were adopted for property determination and material modification. These methods include:

Initial Feedstock Analysis	Method
SARA	In-house, ASTM D6560 (Asphaltene content), solvent used nC7
Sulphur content	In-house
H:C:O (elemental)	ASTM D5291
Inorganic Solid content	In-house, toluene insoluble
Metals content (ICP-MS)	In-house
Simulation distillation	ASTM D7169
Viscosity	In-house using rotational rheometer
TGA Aromatic content	TGA Proximate Analysis: TA-129 by TA Instruments (ASTM D7582)
Softening Point (TMA)	ASTM E2437
SEM (with EDX)	In-house

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

The key results from the feedstock modification activity include:

• Aromaticity and asphaltene content increased upon thermal treatment of vacuum bottom residue where both temperature and duration of the treatment had significant impacts. Treatment temperature and treatment durations both had significant effect on physical and chemical properties of the product. Product viscosity and softening points increased upon thermal treatment of vacuum bottom residue.

Having a high-enough softening point can potentially improve the spinnability of the feedstock material and thus produce filaments with consistent physical properties.

• High coke formation was observed at elevated thermal treatment temperatures. Having coke in the feedstock material will negatively impact its spinnability and more importantly its physical properties.

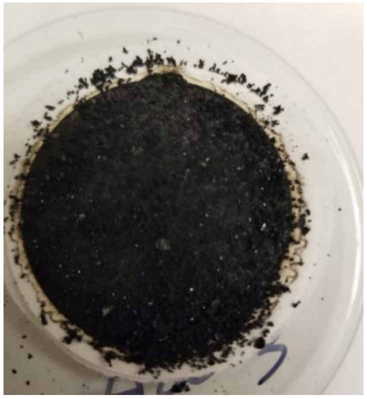

Figure: Toluene insoluble matter obtained after elevated thermal treatment

• To improve material softening point and to potentially enhance aromatic and asphaltene contents, the use of a modified thermal treatment process and incorporating an autooxidation process can be done to achieve this.

• Metal content reduction in the feedstock material can be achieved via solvent extraction process. Reduction of the metal contents in the feedstock material can improve the physical properties of the end product carbon fibres.

The key results from melt spinning the modified feedstock materials include:

• Filaments with diameter in 20-150 µm were generated from thermal treated asphaltenes samples. The filament diameter was influenced by the nozzle diameter and the drum collection speed. The optimum conditions need to be determined to create consistent diameter filaments. Some of the spun filaments were brittle or sticky depending on the feedstock material.

Future Tasks

Future work would explore different spinner nozzle diameters to generate fibres with consistent diameter and investigate the impact of spinner conditions on filament properties. Before the produced carbon fibre physical properties can be determined, the filaments need to be further processed (Oxidation and carbonization). Future work should explore these finishing processes and evaluate the final physical properties on the produced carbon fibre. An iterative approach to feedstock preparation, spinning and carbon fibre formation is necessary to create the desired carbon fibre with appropriate physical properties.

Project Metrics

Metric	Project Target
Pre-treatment methods identified	To explore pre-treatment methods for preparation feedstocks to produce carbon fibre from asphaltenes
Aromaticity	50%
Softening Point	> 220 °C
Spinnability	at lower than 350 °C
Asphaltene Fibre	produced
Metals content	demonstrate that reduction is possible

The procedure to modify the rich in asphaltenes material and create the feedstock for spinning to create filaments was defined. More than 50% of aromaticity was reached. The spinning temperature were less than 350 °C and some filaments were produced form the feedstock prepared. Some exploratory extraction experiments did show the technical feasibility of contaminant reduction. Softening point measurements did not exceed 220 °C, however, that could be due to the technique used and with a different technique it may be above 220 °C.

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

Characterization of asphaltene samples

1. Two feedstock materials were identified from Alberta Innovates' asphaltene sample bank. Overall, there were some differences in the measured values compared to the sample bank data sheet values. This was probably due to variation in sample bank samples. The variations could be due to different sample times, sample original locations, and from different sample providers.

2. Both samples had higher asphaltenes content values compared to sample bank data sheet values. This higher trend was also evident in the carbon content and oxygen content. From the ICP-MS metals analysis, there were a few differences in metal content for the samples compared to the limited sample bank data sheet values. The sample bank data sheet values only had metals content for 8 elements. In our analysis we analyzed 34 different elements.

3. There were also differences in the softening point temperature compared to the sample bank data sheet values. The difference was due to the different ASTM method used to determine the softening point. The values measured in this project were lower compared to the sample bank values. However, in previous studies conducted by ARC, the ASTM method conducted then was the same used in this project.

4. Improvement in material properties is expected to come from thermal treatment and exercising a careful control over S, metal contents and other insoluble present in the material. A high softening point of the material is desirable too.

Feedstock Preparation for Carbon Fibres

1. Aromaticity and asphaltene content increased upon thermal treatment of vacuum bottom residue where both temperature and duration of the treatment had significant impacts. Temperature and time duration appeared to have significant impacts on material aromaticity.

2. Product viscosity and softening points increased upon thermal treatment of vacuum bottom residue.

3. High coke formation (19-21 wt.%) was observed at high temperature. Presence of coke would be detrimental for filament and fibres alike.

4. Improvement of material softening point seems necessary to potentially enhance aromatic and asphaltene contents within the material. Both objectives can be achieved by improving thermal treatment and autooxidation process. A softening point of more than 100°C was achieved by thermal treatment of vacuum bottom residue, a significant improvement over softening point obtained for bottom residue sample. Auto-oxidation steps were able to further improve softening point and aromaticity.

5. Metal content reduction in the feedstock material can be achieved via solvent extraction process. Reduction of the metal contents in the feedstock material can improve properties of the end product carbon fibres.

6. Appropriate feedstocks will need to be spun, oxidized, and carbonized to prepare carbon fibres. Further improvements in feedstock will be required and an iterative approach to feedstock preparation, spinning and carbon fibre formation is necessary.

Spinning

1. No fibres were generated from the precipitated asphaltene sample.

2. Fibres with diameter in 20-150 µm were generated from thermal treated asphaltenes samples at high temperatures. The obtained fibres were brittle and can’t be collected with the drum collector.

3. Fibres were generated from sample at low temperatures. The fibres were sticky and flexible and can be collected with the drum collector.

4. For the next step, spinner nozzle with the diameter of 20µm will be tested to generate fibres with consistent diameter. Due to the brittleness of the asphaltenes based feedstock, modifications on the sample collection part of the spinner may be needed

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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.

Project Outcomes and Impacts

Project outcomes have been able to narrow down the gaps that have challenged the use of asphaltenes-rich feedstocks for carbon fibre making. A few steps to feedstock modification and improvement have been successfully undertaken to this effect. Advancements made have shown some promising results and asphaltene-rich feedstocks with improved material properties have been produced. Improved material properties are desired for spinning of stable filaments. Further improvements in feedstock will be required. An iterative approach to feedstock preparation, spinning and carbon fibre formation is necessary and will need to continue.

Clean Energy Metrics

Metric	Project Target	Commercialization / Mobilization Target
TRL advancement	3	7
Field pilots/demonstrations	0	1
Publications	0	1
Jobs: Acutal new jobs created from project	0	
Jobs: Projected new jobs created from future deployment		10
Patents & Records of Invention filed	1	5
Investment in 4 Core Strategic Technology Areas	$193,000	$10,000,000
New policies informed/influenced	0	0

The target of this project, in terms of Technology Readiness Level TRL, was level 3. Analytical and laboratory study were completed according with the objective defined in the beginning, to prove the concept of modifying the asphaltic material to create an appropriate feedstock to generate later filaments by melting spinning for future carbon fibre. A potential protection or patent could be completed with the experimental procedure or protocol to obtain the feedstock for carbon fibre from asphaltenes.

Program Specific Metrics

Metric	Project Target	Commercialization / Mobilization Target
Unique product/process	1	3
# commercial BBC products	0	1

A unique process to treat asphaltenes is under development because of this work. Success in future work will lead to multiple products being generated from this process.

Project Outputs

There is no doubt of the relevance of the information generated here for future publications and conferences. But the confidential character of this work, at least at this stage, force InnoTech Alberta to maintain the data and results under the custody of AI. Future external communications will be prepared for public diffusion at the next stages coming.

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

The key benefits to the knowledge generated from this project includes:

Economic

The future manufacture of carbon fibre from asphaltenes in Alberta will attract local and external investment with the generation of jobs and opportunities for the province. The interest for the utilization of this material to produce others material and composite for different uses will also impulse industrial and manufacture activities. Today is early to see the benefits after the conclusion of this project. More technical work is required to complete the proof of concept and move finally to the pilot and industrial stage. A new opportunity of getting better returns for low-cost bitumen product will likely help to attract investments from Alberta oil producers for development of carbon fibre technology.

Environmental

The environmental benefits are going to be visible later with the manufacture of light and more resistant materials for industries like the construction and vehicle manufacture. Continued improvements to technical know-how for production of carbon fibre in Alberta using asphaltenes will open the possibility of having light and resistant material to reduce the combustible consumption. Reduction of waste streams of Alberta oil producers is one of the other aspects to be considered.

Social

Commercial uses of asphaltenes as feedstock for value-added product will also place the bases for the generation of new jobs related with the industrialization of applications of Alberta carbon fibre.

Building Innovation Capacity

The project gave to professionals and technologist the chance to acquire a fundamental and technical knowledge in this area. Some equipment and techniques were installed and tested to modify the asphaltenes and create the filaments. Spinners to process and form the filaments from this novel material were used and an important expertise was obtained for the new initiatives coming including the future pilot and industrial facilities. The setup and the knowledge are available to other organizations with the interest of developing this new way to use waste materials from the Alberta bitumen.

I. RECOMMENDATIONS AND NEXT STEPS

The following are the recommendation for 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

- Continue to expand on the initial work to create a stock of spinnable feedstock material that can produce carbon fibre with the desire physical properties. The feedstock modifications done to date show the improvement of feedstock properties. However, the best feedstock conditions have not yet been identified. This work needs to be an iterative approach where the feedstock material is spun and processed to create the carbon fibres, the properties are then determined, and the results are fed back to the feedstock modification conditions.
- Potential other starting feedstock material could be explored. This project only considered two original feedstock materials from Alberta Innovates' sample bank.
- Explore the filament spinnability conditions to generate consistent filaments. There are a number of melt spinning process conditions that need to be explored further to develop the best spinning conditions.

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

The technical information from this initiative will be divulged on meetings with the different industrial sectors: oil producers, carbon fibre producers and end user of the carbon fibre. Seminars, technical presentations and also guided visit to the InnoTech Alberta laboratories will be organized to show the systems and apparatus during the operation, from where the materials were modified and after processed to produce the filaments, and the techniques for the characterization. Also, the high temperature ovens for the filaments processing and the formation of carbon fibre from asphaltenes will be presented.

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

The following are the main conclusions from this project:

- Aromaticity and asphaltenes content increased upon thermal treatment of vacuum bottom residue where both temperature and duration of the treatment had significant impacts.
- Product viscosity and softening points increased upon thermal treatment of vacuum bottom residue. Having an improved softening point can potentially improve the spinnability of the feedstock material and thus produce filaments with consistent physical properties.
- High coke formation was observed at elevated thermal treatment temperatures. Have coke in the feedstock material will negatively impact its spinnability and more importantly, its physical properties.
- To improve material softening point and to potentially enhance aromatic and asphaltene contents, the use of a modified thermal treatment process and incorporating an autooxidation process can be done to achieve this.
- Metal content reduction in the feedstock material can be achieved via solvent extraction process. Reduction of the metal contents in the feedstock material can improve the physical properties of the end product carbon fibres.

The key results from melt spinning the modified feedstock materials include:

- Filaments with diameter in 20-150 µm were generated from thermal treated asphaltenes samples. The filament diameter was influenced by the nozzle diameter and the drum collection speed. The optimum conditions need to be determined to create consistent diameter filaments. Some of the spun filaments were brittle or sticky depending on the feedstock material.