

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 <u>is</u> <u>free of any confidential information or intellectual property requiring protection</u>. 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.



CLEAN RESOURCES FINAL PUBLIC REPORT TEMPLATE

1. PROJECT INFORMATION:

Project Title:	A Canadian Sustainable Solution for the Production of Carbon Fibre
Alberta Innovates Project Number:	G2020000344
Submission Date:	2021-02-21
Total Project Cost:	\$100,000
Alberta Innovates Funding:	\$50,000
Al Project Advisor:	Murray Gray

2. APPLICANT INFORMATION:

Applicant (Organization):	McGill University
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3. PROJECT PARTNERS

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

RESPOND BELOW

Prof. Milan Maric, Prof. Reghan Hill, Prof. Chao-Jun Li

A. EXECUTIVE SUMMARY

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

RESPOND BELOW

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 project, minimal designer-ionic liquids was used to successfully dissolve asphaltene samples in solid and liquid phase. The material obtained was thermally stable and further optimization of the rheological properties will enable the continuous carbonization to 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.

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:

Carbon fibre (CF) is a material that contains more than 92% of carbon. The approach utilized today to produce CFs involves many steps with proprietary processes secretly kept by the CFs producers around the world (None in Canada). Carbon fibre production starts from the spinning of a precursor fibre (rayon, pitch, and polyacrylonitrile (PAN)) from crude oil cracking. Most CFs (96%) are produced from PAN that are pretreated, oxidized and carbonized to obtain different grades of fibres. Carbon fibres are then combined mostly with polymers to form advanced composites mostly for the aerospace,

transportation, energy and sporting industry. The worldwide demand for CF is estimated at 142,000 tonnes in 2020 with an average compound annual growth rate superior than 13%. The production of CFs is dominated by about 15 manufacturers located in North America (none in Canada!), Europe and Asia. Making CFs requires very high capital costs with very high barrier to enter their production using the conventional precursor to fibre process. The CFs value chain starts with crude oil (\$0.72/kg based on the barrel at \$100) to the PAN precursor (\$3-6/kg) and finally low-grade CFs (\$30/kg) or high-grade CFs (\$100/kg). Obviously, getting into the game of making CFs requires a new innovative approach specifically adapted to the Alberta oilsands industry.

Knowledge or Technology Gaps:

The Alberta petroleum production, mostly oriented to combustion applications, produces a significant amount of asphaltene that can be used to make CFs. A patent in that effect was filed by Honeywell Federal Manufacturing & Technologies in 2017 (US 9,580,839 B2). The claim is the extraction of the asphaltene from the coking reactor and through a series of filtration steps, an asphaltene fibre is spun before entering a carbonization process. It is not clear from this patent what are the properties of the carbon fibre obtained and if that approach can be adapted to the Alberta oilsands asphaltene feedstock. Currently, CF production from oilsands asphaltenes are mainly by two methods: 1) Use low polar volatile solvents such as hexane and toluene to extract asphaltenes and then produce a fibre with concurrent evaporation of solvent followed by high temperature and oxygen-free carbonization; 2) after generating asphaltenes through the above extraction, the asphaltenes are then mixed with other polymers to generate a composite fibre and then carbonized at high temperature under oxygen-free conditions. The main challenges of the current methods are two-fold: 1) only a small portion of bitumen carbon material can be used due to the low solubilizing power of hexane and toluene towards asphaltenes, and 2) the carbonization process is batch-based and relatively slow.

The proposed solution must then seek to produce CFs from asphaltene in a more efficient and sustainable way. We have to take advantage of the large availability of low cost asphaltene and low cost clean electricity in Alberta. Combining the scientific expertise in polymer chemistry and in advanced composites across Canada, our team wants to develop a new approach to make CF that is distinct from what is done today. We believe that taking a high-risk approach is paramount to develop a Canadian CF

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

Updates to project objectives: In this project, minimal designer-ionic liquids were used to successfully dissolve asphaltene samples in solid and liquid phase. This approach minimizes wastes and has the potential to significantly reduce the energy required to produce carbon fibres.

Performance Metrics: The project aims to train 2 Post-Doctoral Fellows, to sign one partnership agreement with a partner in Alberta and to identify one collaboration partner toward Phase II. The project success metrics are that asphaltene is soluble in ionic liquid and that the asphaltene/ionic liquid rheologic properties are in range for fibre spinning.

Milestones 1 and 2 were successfully completed, Milestones 3 and 4 were not undertaken as the dissolution of the asphaltenes in ionic liquids was more challenging than expected, and delays due to COVID 19 reduced the scope of work that could be completed.

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

In this work, eight minimal designer-ionic liquids (ILs) were selected to dissolve the solid asphaltene sample S2 provided by the asphaltene bank. Small sample of solid asphaltene were mixed using a range of methods, under different atmosphere and temperature. Various concentrations of ILs were used and the effect of solvents was also investigated. The asphaltene-ILs mixtures were tested for their thermal stability using thermogravimetric analysis. The mixtures rheological properties were investigated using a parallel plate rheometer. Strain and frequency sweep procedures were used to extract the rheological parameters relevant to fibre spinning.

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

Asphaltene samples with 30-50% Ionic liquid content were synthesized. The samples were thermally stable to temperatures up to 225° C and started to degrade at temperatures over 300° C. The samples behave as a shear thinning fluid, but currently exhibit low elasticity to spin fibers with them. Their yield stress was in the range of 0.4 - 84 Pa and their flow stress ranged from 0.92 - 25 Pa. Two of the eight ILs tested showed promising results. The mixing method dispersed better the asphaltene in the ionic liquid. The thermal stability analysis shows better homogeneity for the sample mixed with the mixing technique.

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

Asphaltene particulates are wetted and dispersed (to varying extents) in ionic liquids. The rate of dissolution is enhanced by the mixing method. Dissolution may be limited by mixing time and the temperature being below an upper critical solution temperature (UCST). Thermal stability limits have been established by thermogravimetric analysis. Dispersion rheology was measured, exhibiting shear-thinning. Asphaltene dispersions that flow as liquids can be prepared.

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

This project enabled the training of three highly qualified personnel on the topic of asphaltene/ionic liquid mixtures. Discussion with one Alberta partner was initiated to continue the work on the characterization of asphaltene ionic liquid mixtures. A potential partner for Phase II was identified.

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

Not applicable for this project.

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

In term of technical next steps, an upper critical solution temperature (UCST) should be determined, to provide optimal dissolution conditions (time and temperature). Optical and electron microscopy should be used to establish the maximum asphaltene loading (per unit mass of ionic liquid) that produces asphaltene solutions, ruling out asphaltene particulate dispersions. A method should be developed to dispense asphaltene solutions/dispersions into a high-temperature oxygen-free environment to promote graphitization. A method should be developed to extrude and/asphaltene solutions/dispersions.

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

There are not plans at this stage to disseminate the results from this project.

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

In this project, minimal designer-ionic liquids were used to successfully dissolve asphaltene samples in solid and liquid phase. The material obtained was thermally stable and further optimization of the rheological properties will enable the continuous carbonization to produce carbon fibres from the ionic liquids/asphaltene mixture.