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 AL PUBLIC REPORT TEMPLATE

1. PROJECT INFORMATION:

Project Title:	Towards Large-Scale Unconventional Micro-meter Diameter Carbon Fiber Production
Alberta Innovates Project Number:	G2020000335
Submission Date:	18 March 2021
Total Project Cost:	68 000 CAD
Alberta Innovates Funding:	50 000 CAD
AI Project Advisor:	Murray Gray

2. APPLICANT INFORMATION:

Applicant (Organization):	University of Alberta
Address:	DICE building, Office, 10-360
Applicant Representative Name:	Cagri Ayranci
Title:	Associate Professor
Phone Number:	(780) 298-0518
Email:	cayranci@ualberta.ca

3. **PROJECT PARTNERS**

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

RESPOND BELOW

We did not have a partner 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

This project looked at the manufacturing feasibility and viability of carbon fibers from AB based asphaltene obtained from AI. Our group explored a variety of manufacturing processes and converged on the successful production of short carbon fibers through an innovative manufacturing process. We are in the process of filing an ROI at the U of A. We generated carbon fibers using Asphaltene Sample S2. Our precursor fibers are composed of Asphaltene and an aiding polymer. The generated carbon fibers ranged from 1-3 µm in diameter. The produced fibers showed favorable traits such as alignment, separation between each fiber. Furthermore, they can be produced in length varying from 5-50 mm without the requirement of chopping or cutting.

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:

Short carbon fibers can be used in a variety of applications, including concrete and reinforced concrete for civil engineering applications, electromagnetic interference and a series of electronics such as battery production, and solar panel production. At the moment, short carbon fibers are generally produced by creating long carbon fibers and chopping them to the desired length.

Knowledge / Technology Gap:

Fiber production with recycled asphaltene is challenging due to the impurities presented in the material. But the low cost of the Asphaltene as a precursor, compared to the conventional expensive carbon fiber precursors, makes it an attractive alternative. A manufacturing method that is scalable and simple to produce was developed to manufacture short carbon fibers.

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 the project consisted on the production of carbon fibers via electrospinning and melt-electrospinning. Both of these techniques were investigated but the results showed a problematic manufacturing process difficult to scale up. Although fibers before carbonization were produced with both methods, the quality of the fibers were not appropriate for industrial applications. Instead, a new manufacturing process for the production of short carbon fibers was proposed. The proposed manufacturing process is of low power consumption, fibers do not require post processing to create short carbon fibers, the fibers can be easily separated and collected aligned or randomized, and is possible to create different mats at different angles with this technique. The manufacturing process showed great potential as it can be easily scaled for industrial production. The manufactured fibers showed consistent fiber diameter along the length of the fibers with diameters between 1-3 μm.

Performance metrics have changed since the submission of the original project. As measurement of mechanical properties of the short fibers require a longer and tedious approach, due to the COVID closures, this task was not done in Phase 1.

D. METHODOLOGY

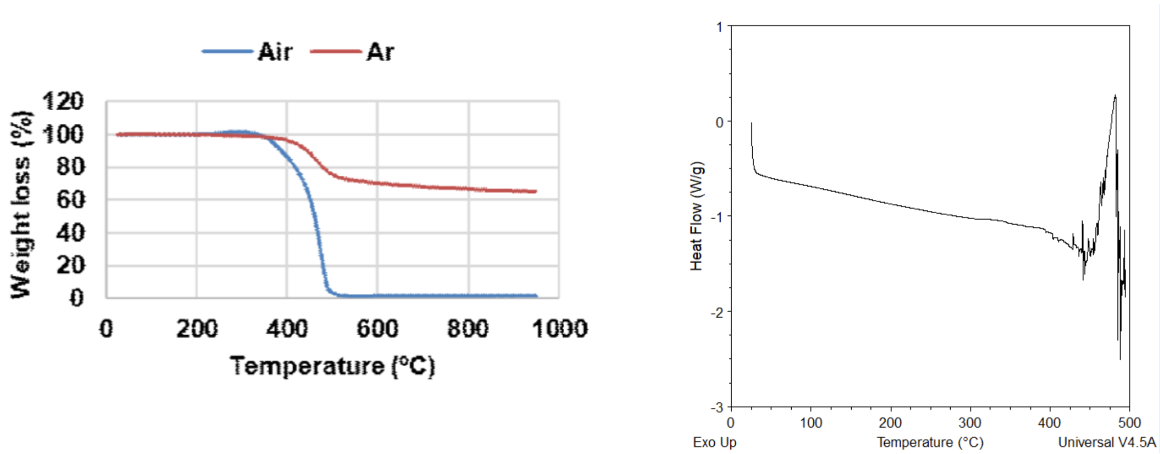
Please provide a narrative describing the methodology and facilities that were used to execute and complete the project. Use subheadings as appropriate.

RESPOND BELOW

All the experiments and characterization is done at the PIs laboratories as well as at fee for services facilities at the University of Alberta. The method utilized to produce the fibers will not be explained here due to our ROI filing at the U of Alberta. However, following are the methodologies used to characterize the materials.

Material Characterization:

Material characterization of Asphaltene Sample S2 provided by Innotech Alberta was conducted. Thermogravimetric Analysis (TGA) (Figure 1(a)), Differential Scanning Calorimetry (DSC) (Figure 1(b)), and elemental analysis were conducted. From the TGA analysis (Figure 1(a)), the batch showed a degradation temperature at approximately 350 °C in air and 450 °C in Argon. The DSC in air test showed a softening point at approximately 300°C.



(a) (b)

Figure 1. (a) TGA and (b) DSC results of the material characterization.

The conducted elemental analysis showed similar results to those provided by Alberta Innovates. The results can be observed in Table 1.

Table 1. Elemental Analysis comparison from those provided by Alberta Innovates and University of Alberta.

	%C	%H	%N	%S	%O
AI	83.3	5.7	1.7	7.1	1.5
U of A	83.7	6	1.6	7.2	<2

Material solubility was qualitatively characterized by trying different types of solvents as shown in Table 2. Solubility was best obtained in Toluene and THF.

Table 2. Material solubility

Solvent	Solubility

Toluene	**Soluble**
Benzene	**Partially soluble**
Methylene chloride	**Partially soluble**
Pentane	**Insoluble**
THF	**Soluble**
DMF	**Slightly soluble**

Fiber Production

During the project, different manufacturing techniques were explored with the aim to achieve the least cost and high performance solution for Asphaltenes fiber manufacturing. Asphaltenes Sample S2 was provided from Innotech. The batch was purified using ASTM D2007. Residuals of the impurities unsolvable in Toluene can be observed in Figure 2.

Figure 2. Insoluble residuals found in asphaltene after the cleaning process

Different ratios of Asphaltene and **[aiding polymer name deducted]** are dissolved in DMF and Toluene to form solutions to produce fibers containing (0, 18, 45, 50 and 60 % Asphaltenes). A method of manufacturing the short fibers was developed using these blends.

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

E. PROJECT RESULTS

Production of Short Fibres from Asphaltene Polymer Blends

Photographs of different compositions of produced fibers can be observed in Figure 3.

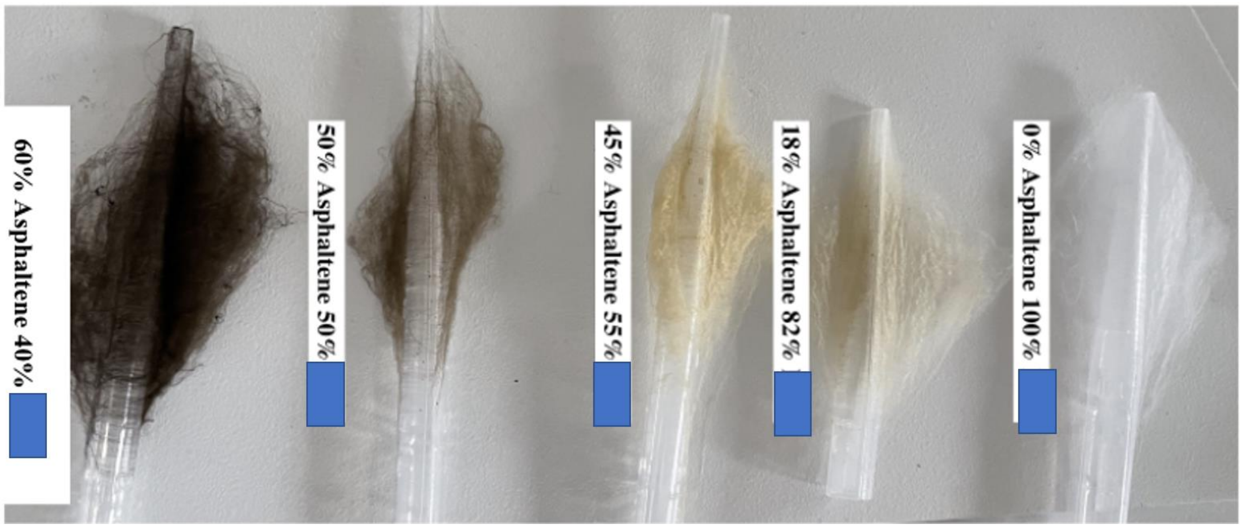


Figure 3. Manufactured short fibers showing a decreasing amount of asphaltene content in the manufactured fibers.

The fibers were able to be collected in a randomized manner and in an aligned manner. The manufacturing technique enables the ability to manufacture aligned mats in different angular configurations, such as [0,90] degree plies.

Stabilization and Carbonization of Fibres

Asphaltene fibers before thermostabilization are presented in Figure 4. Figure 4.a depicts Asphaltene fibers as collected and stretched fibers before thermostabilization are shown in Figure 4.b.

(a) (b)

Figure 4. 60% Asphaltene fibers manufactured in aligned configurations with (a) the fiber mat collected and (b) stretched fibers before thermostabilization.

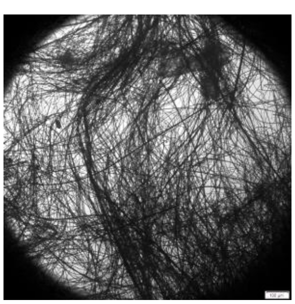
The fibers were thermostabilized at 0.25°C/min to 140°C and heated isothermally for 3 hrs, followed by a heating ramp of 0.5°C/min to 300°C and heated isothermally for 2 hrs. The Fibers were then carbonized by increasing the temperature to 500°C at 2°C/min and held for 1 hr, followed by a heating ramp of 2°C/min to 800°C and held for 1 hr. The fibers were carbonized at 800°C and 1000°C. Photographs were taken of fibers before and after carbonization and are shown in Figure 5 .

(a) (b)

Figure 5. Photographs of produced fibers (a) before carbonization (weight 0.0142g) and (b) after carbonization (0.0057g)

Properties of Carbonized Fibres

Results of fiber production are presented in this section. The produced precursor fiber diameter ranges from 1-3µm. An optical microscopy of the obtained fibers shows consistent diameter throughout the length of the fiber. as observed in Figure 6. A DSC result of fibers before carbonization can be observed in Figure 7. The results show a melting point of **[aiding polymer name deducted]** at around 110 °C and degradation starting at 400 °C.



(a) (b)

Figure 6. Microscopy of produced fibers before carbonization (a) photograph of fibers before carbonization.

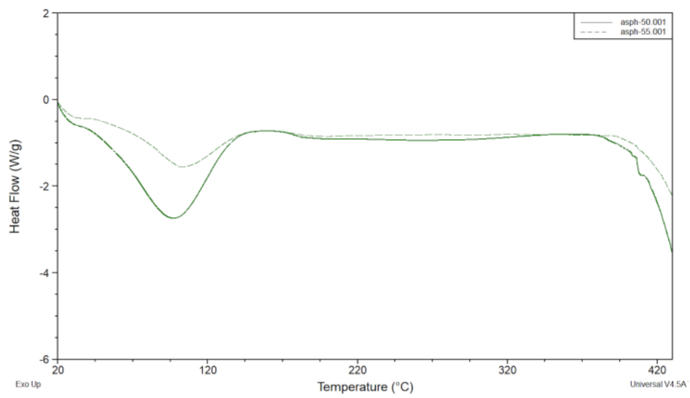
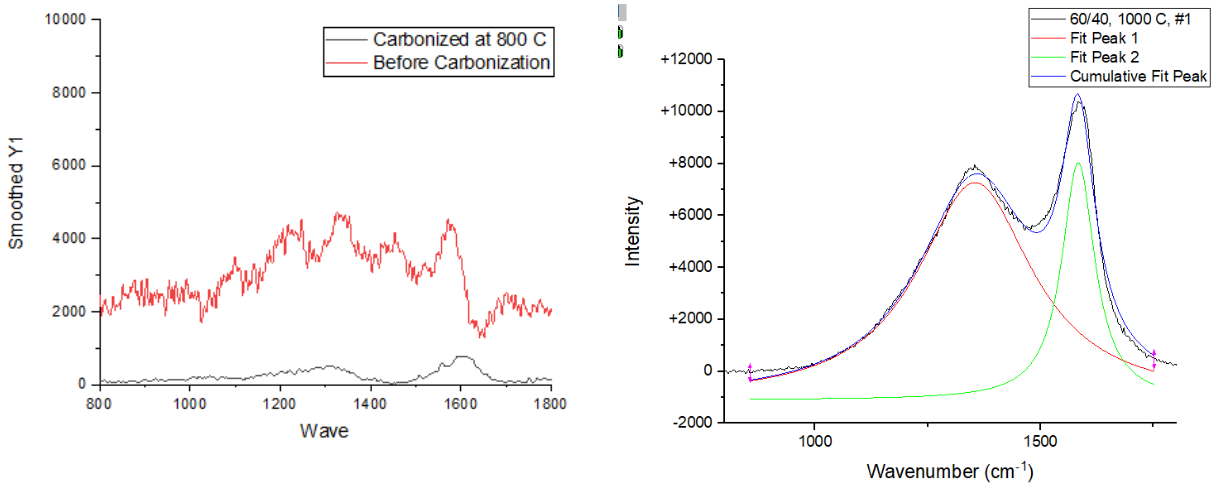


Figure 7. DSC of fibers produced with different asphaltene

Carbon fibers were obtained after carbonization at 800 and 1000℃ for different samples. A photograph of fibers carbonized at 1000℃ is shown in Figure 8. The carbonized material was characterized using Raman spectroscopy. Figure 9. displays the Raman spectrum of the carbonized fibers (a) and its deconvolution (b) before and after pyrolysis. The spectroscopy shows the generation of peaks after carbonization has occurred. This indicates the formation of ordered and disordered carbon structures. A sample of the spectroscopy for 1000℃ can be observed in Figure 9.b.

Figure 8. Figure of carbonized fibers at 1000℃



(a) (b)

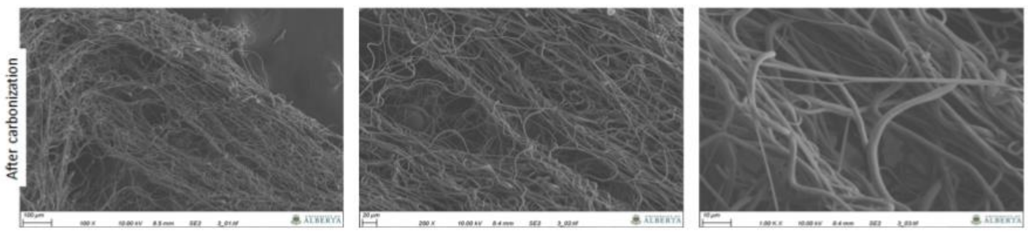
Figure 9. Raman spectroscopy of (a) fibers before and after pyrolysis for fibers carbonized at 800 °C and (b) for fibers carbonized at 1000 °C.

The elemental analysis conducted on the obtained carbon fibers showed a carbon fiber content of approximately 71.5%. Further investigation to enhance carbon content needs to be conducted. The complete elemental analysis can be observed in Table 3.

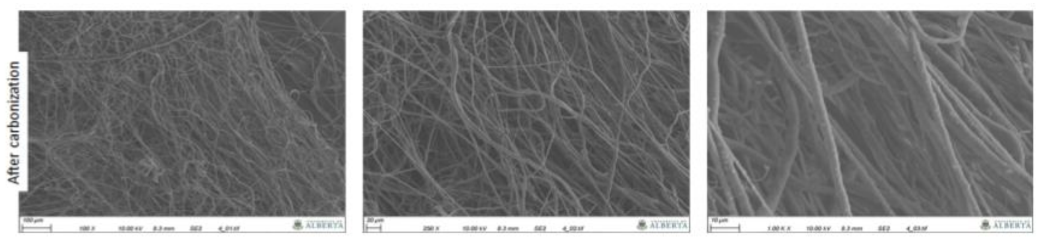
Table 3. Elemental analysis of carbon fibers carbonized at 1000℃

Sample	Wt. (mg.)	%N	%C	%H	%S
60-40	1.4869	4.871	71.456	1.939	2.661

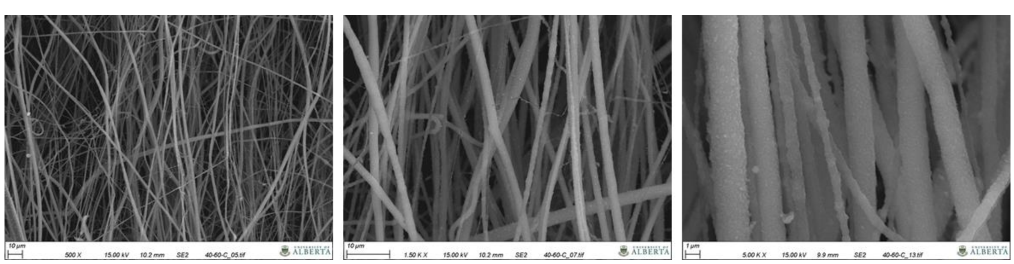
Fiber morphology did not change before and after morphology observation. In the following figure, the structures of 50%, 55% and 60% asphaltene content after carbonization can be observed. 50 and 55% were collected in a non aligned manner, while 60% asphaltene fibers were collected in an aligned manner. There is a roughness to the fibers that needs to be further analysed and investigated.



(a)



(b)



(c)

Figure 10. Carbon fiber morphology with (a) 50%, (b) 55%, (c) 60% Asphaltenes content.

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

Our group produced successfully short carbon fibers with an innovative manufacturing process. The manufacturing process is efficient compared to the current techniques used in the industry. Manufactured fibers showed flexibility and approximately 71% carbon content. Our manufacturing process can produce short fibers of diameter of 1-3 µm. Our manufacturing process is simple in concept, can produce small fibers with small power consumption, tailor the fiber properties and produces a small amount of waste.

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.

Outcomes and impacts:

As aforementioned, a new short fiber carbon fiber production technique for asphaltene based precursors has been developed.

Clean energy metrics: Due to the aforementioned reasons (change in methodology as well as COVID limitations) some changes was done. One ROI is in the process of being filed. This will be followed by a journal paper submission. 2 PhD and 1 PDF worked in the project.

Program specific metrics:

Due to the change in nano CF to short CF production in the target product, our end target companies that we will approach has changed. Our unique product is asphaltene based short CF fibers.

Project outcomes:

1 ROI is in the process of filing. This will be followed by a journal paper. Due to the 6 month period, the work did not result in any thesis.

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.

Economic:

N/A for Phase 1.

Environmental:

N/A for Phase 1.

Social:

N/A for Phase 1.

Building Innovation Capacity:

2 PhD students and one PDF has worked on the project. Their learnings as HQP is invaluable to their professional development. The development of the new CF production technique as part of the project was a great opportunity for the HQP to appreciate the importance of methodological study and isolation and investigation of fundamental phenomenon in multidisciplinary scientific research projects.

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

Throughout the project our group encountered great potential to manufacture short carbon fibers easily and in a controlled manner. However, the technology is still in the developing stage and an automated version of the developed technology needs to be put forward. This technology is filed as an ROI towards a patent. Further investigation after the technology has been automated will need to be conducted to improve fiber roughness, control fiber diameter and decrease material waste. In terms of performance, electrical, mechanical and thermal properties will need to be further investigated to achieve potential applications in the construction, automotive and electronics sectors. As such, related industries will be contacted for partnering for Phase-2.

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

Our group produced successfully short carbon fibers with an innovative manufacturing process. Manufactured fibers showed flexibility and approximately 71% carbon content. Our manufacturing process can produce short fibers of diameter of 1-3 µm. Our manufacturing process is simple in concept, can produce small fibers with small power consumption, tailor the fiber properties and produces a small amount of waste.

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

Asphaltenes were obtained from Alberta Innovates. The asphaltene was purified and multiple manufacturing processes were explored. We converged on a simple manufacturing process. The proposed novel manufacturing method is of low power consumption, fibers do not require post processing to create short carbon fibers, the fibers can be easily separated and collected aligned or randomized, and is possible to create different mats at different angles with this technique. The manufacturing process showed great potential as it can be easily scaled for industrial production. The manufactured fibers showed diameters between 1-3 µm. Different asphaltene contents were investigated to optimize the asphaltene content in the solution. Different thermostabilization and carbonization temperatures were tried to create flexible carbon fibers. Carbon fibers were manufactured by stabilizing the produced fibers at 140°C and finally carbonizing them at 1000°C. The fibers were characterized by Raman spectroscopy, and by characterizing the morphology of the fibers through SEM images. A consistent diameter was observed throughout the length of the fibers, at the moment the fibers do show roughness in their surface. Further improvement in carbon content, surface improvement and mechanical properties will need to be addressed in a future work.