Carbon Fibre from Alberta Oilsands Asphaltenes (AOA)

Public Report

Prepared for

Alberta Innovates

Prepared by

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ALBERTA INNOVATES

CLEAN RESOURCES FINAL PUBLIC REPORT TEMPLATE

1. PROJECT INFORMATION:

Project Title:	**Carbon Fibre from Alberta Oilsands Asphaltenes (AOA)**
Alberta Innovates Project Number:	G2020000342
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AI Project Advisor:	Paolo Bomben

2. APPLICANT INFORMATION:

Applicant (Organization):	**Deakin University**
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3. PROJECT PARTNERS

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

RESPOND BELOW

A. EXECUTIVE SUMMARY

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

RESPOND BELOW

Canada contains the world's third largest proven oil reserve and ~ 97% of these are present in the form of oil sand. Canadian oil sand is the mixture of sand, water, and bitumen. Asphaltene is one of the compounds, retrieved from bitumen and available abundantly around Canadian oil reserves. Therefore, it is important to find a value-added application for such asphaltenes. To investigate the possible relevance, the intent of this project is to develop a carbon fiber from Alberta oil sand asphaltene (AOA). Currently, the global carbon fiber market is largely dominated by polyacrylonitrile (PAN), and it is major contributor to the higher cost of carbon fiber. Thus, developing carbon fiber from asphaltene can be an eminent alternative to low cost carbon fiber. In general, carbon fiber from asphaltene is developed by converting them into mesophase pitch followed by meltspinning. In order to contemplate the possibility of producing carbon fiber from asphaltene, two simultaneous strategies have been employed: (i) chemical and thermal modification to find the possibility of melt-spinning AOA (ii) blending AOA with PAN for wetspinning precursor fiber followed by converting them into carbon fiber. The chemical modification of AOA was carried out by the conventional chemical extraction method. The asphaltene modification were also carried out by ionic liquid in addition to ultrasound assisted extraction. Apart from chemical modifications, Alberta asphaltenes were also modified thermally. The preliminary observation of AOA modification has revealed that the asphaltene extracted from conventional chemical modification solvent mixture of n-hexane/n-heptane was thermally more stable compare to all other extracted method. However, meltspinning trials of any modified samples were not successful due to presence of high insoluble content with a wide molecular weight distribution. Nevertheless, the initial study of meltspinning process demonstrated the potential of asphaltene to be converted into carbon fiber via meltspinning process. This calls for further investigations into meltspinning process in future and therefore in the interest of project deadline, it was decided that the focus shifts on the development of carbon fiber via wetspinning method.

Carbon Nexus at Deakin university house the unique facility of converting range of precursor fiber into carbon fiber. This facility involves three different setups including carbon fiber simulators for smaller tow size ≤3k, research line and pilot line with the capacity of 3k to 320k tow size. Since this project is focused on the feasibility study of AOA based carbon fiber, we have exploited the carbon fiber simulator for converting spun precursor fiber into carbon fiber. The process for converting wetspun fiber into carbon fiber includes stabilization followed by carbonization. The stabilization process converts the general structure of PAN into non-degradable, non-flammable ladder like structure by virtue of cyclization, dehydrogenation, and oxidation. In general, Carbonization process was performed on stabilized fiber in the temperature range of 400℃ to 1400℃ in inert (N2) environment. The current project involves the standard stabilization and carbonization process with some degree of optimization and significant processing modification due to the presence of asphaltene which exhibited different attributes during the entire spinning, oxidation, and carbonization process. The obtained carbon fibers have resulted in tensile strength of more than 700 MPa and modulus more than 150 GPa. For higher wt% of AOA, the properties were found to be reducing (tensile strength ≤400 MPa and modulus <130 GPa) but mechanical properties of developed carbon fiber show the potential of converting asphaltene into value added carbon fiber application. It is important to note here that the final mechanical properties of carbon fiber are not governed by just simple terms of wetspinning, stabilization and carbonization. In fact, the entire process contains significant number of parameters and thus the carbon fiber with acceptable commercial properties needs every parameter to be optimized. The application of carbon fiber is not limited to the automotive or aerospace, it can be also utilized for many other applications like flame proof structure, medical and acoustic applications etc. Therefore, the current project can be a significant step towards the development of carbon fiber from an alternative source, referred to as asphaltene.

Contents

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

At the opening of Alberta's first commercial oil sands plant near McMurray, Alberta in 1967, J. Howard Pew stated that "No nation can long be secure in this atomic age unless it be amply supplied with petroleum . . . It is the considered opinion of our group that if the North American continent is to produce the oil to meet its requirements in the years ahead, oil from the Athabasca area must of necessity play an important role". From the start of 1967, Canada now contains third largest proved oil reserve in the World, which is 169.7 billion barrels at the end of 2019. Canada began the oil production by 12000 barrels/day in 1967 and now reached to the daily production of 5.65 million barrel per day in 2019 and expected to grow further. Importantly, these oil resources in Canada are found mostly in the form of oil sands in the region of Athabasca, Peace River, and Cold Lake deposits in Alberta and Saskatchewan. The Canadian oil sand is the mixture of sand, water, clay and bitumen containing API gravity of less than 10^{o} and viscosity more than 10,000 cP at reservoir condition as shown in Figure 1(a). This is regarded as the most viscous hydrocarbon and mostly coined as bitumen. Primarily, the bitumen content in oil sand defines the typical class of oil deposits such as low-grade oil sand (6-8% of bitumen), medium grade (8-10wt% bitumen) and rich grade (>10wt%). The general assessment of Alberta oil sand bitumen reveals that its composition involves 15-20 vol% of asphaltenes, 45-50 vol% of resid, 35-40vol% of vacuum gas oil (VGO) and 15-20 vol% of distillate. The high viscosity of the bitumen is associated to the presence of asphaltene which is defined based on its solubility in solvents. Asphaltenes are primarily soluble in solvent 6 or benzene and but insoluble in lower alkanes, such as n-pentane or n-heptane as illustrated in Figure 1(b). Asphaltenes are polar component with high aromacity, molecular weight, density, heteroatom and metal content. The properties of asphaltenes are critically dependent on their constituents which largely changes with the source of origin. Asphaltenes are generally considered a threat in the oil field due to their ability to increase the bitumen viscosity but it also finds some commercial applications such as road constructions, water proofing, corrosion inhibitors etc. Apparently, the presence of high polycyclic aromatic hydrocarbons in asphaltenes is also gleaned to be a potential precursor for the development of functional carbon materials like carbon foam, active carbon, carbon fiber etc. Among various applications, carbon fiber is found to be the most promising and value-added application of asphaltene.

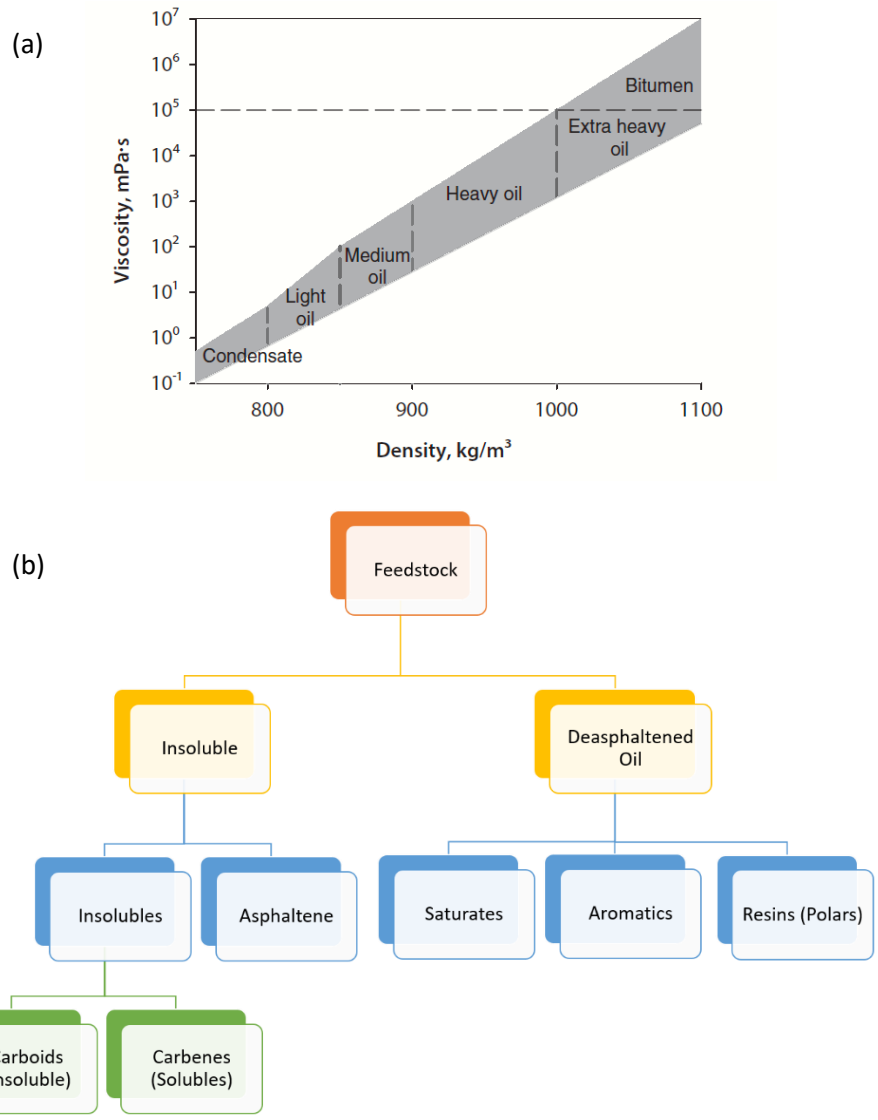


Figure 1 (a) Viscosity and density of crude oil and bitumen (b) Representation of separation of various fraction from petroleum oil

Carbon fiber is high strength and high modulus engineering fiber containing at least 92% of carbon. The mechanical properties of carbon fiber largely vary by number of factors including type of precursor materials (polyacrylonitrile (PAN), mesophase pitch, cellulose and Rayon), method of production, processing techniques and parameters etc. Although, the properties of carbon fiber are stimulated by many manufacturing and processing techniques, but precursor materials play decisive role in their final tensile strength and modulus as illustrated in Figure 2. Among various precursor materials, polyacrylonitrile dominates the carbon fiber market and covers approximately 90% of the market due to their range of mechanical performance and carbon yield. However, high cost of manufacturing PAN based CF is a major concern for exploiting the full potential of the carbon fiber. Approximately, 51% of the total cost is associated to the PAN precursor. Therefore, extensive efforts have been employed in order to reduce the precursor cost and many other alternatives have been examined in the recent years but

commercially achieving the minimum required mechanical properties (DOE target- Tensile strength~1.7GPa, Tensile Modulus~170GPa) is yet to materialize for applications like automobiles.

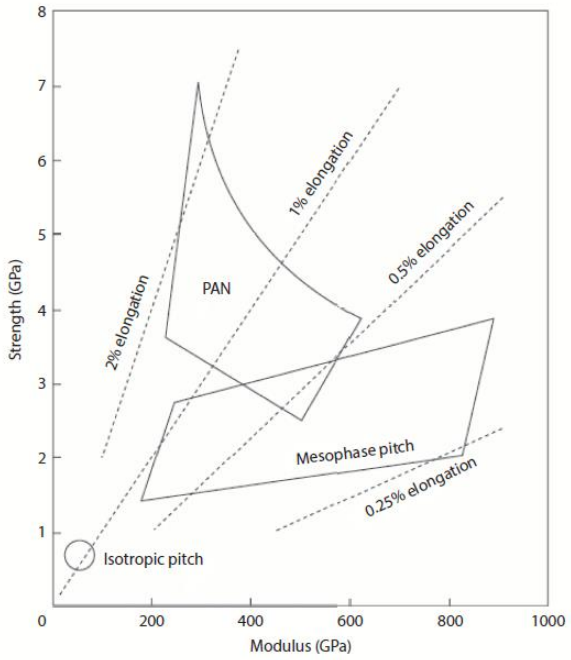


Figure 2 Mechanical properties of carbon fiber obtained from various precursor (P. Morgan, Carbon fiber and their composites, CRS Press, 2005)

Asphaltene from Alberta oilsands (AOA) is polycyclic aromatic hydrocarbons containing high mass fraction of carbon (~83%). The high carbon content available in AOA may facilitate the development of multifunctional active carbon-based materials and carbon fiber can be one of them. To develop AOA as alternative low cost precursor for carbon fiber two distinct approaches can be employed i.e. directly spinning into the fiber by utilizing available spinning method and subsequently converting into carbon fiber or blending (mixing) the alternative precursor with polymer to reduce the requirement of PAN precursor in the system and subsequently saving the cost. The former approach of converting AOA into precursor fiber requires melt spinning method. The preliminary assessment of asphaltenes have revealed that it cannot be directly converted into carbon fiber due to broad range of softening-melting point, less degree of polymerization and inadequate rheological properties. As reported, asphaltene must be thermally condensed or chemically modified to obtain spinnable pitch with mesophase like features. The commercial viability of the carbon fiber can be only realized when AOA is converted into mesophase pitch. Petroleum-based asphaltene (may be applicable to AOA too) contains low aromacity, higher molecular distribution compared to coal based asphaltenes, which hinders melt spinning and subsequent conversion into carbon fiber. To avoid the additional processing methods, AOA can be blended or mixed with PAN to reduce the requirement of precursor material in the system and subsequently save the cost of the production. Table 1 represents the comparative study of both the approaches and demonstrate the possible method to convert AOA into carbon fiber. Both the process of meltspinning and wetspinning have their own advantages and disadvantages. Therefore, selection of suitable process for converting AOA into carbon fiber is essential.

Table 1 Comparative table of meltspinning and blending PAN AOA with PAN

Melt Spinning AOA	Blending AOA with PAN
Requires chemical and thermal pretreatment since asphaltene in pristine form cannot be melt spun	Can be directly used with PAN
To achieve required properties needs additional graphitization (~3000℃) stage	Can be processed with similar condition required for PAN
Commercially available tow size for pitch is 16k i.e. asphaltene derived pitch-based CF can be produced in limited tow size	Commercially available size is 60k and found even in higher tow size

To adapt any of the above stated approach, it is important to assess the thermal and chemical properties of as received AOA. Therefore, the project primarily focused on initial evaluation of pristine AOA and further assessing the possibility of developing precursor fiber by utilizing either meltspinning or wetspinning, given the 6 months duration of the project. To obtain the carbon fiber, the developed precursor fiber will be oxidized followed by carbonization process. The project also involves the characterization of obtained carbon fiber for evaluation of its mechanical properties via single fiber filament testing as well as scanning electron microscopy for study of fiber surface morphology and RAMAN analysis.

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

TECHNOLOGY DESCRIPTION AND PROJECT

The project aims to explore conversion of oilsands asphaltenes into carbon fiber. To the best of our knowledge and experience, direct conversion of asphaltenes to carbon fibers has never been reported, neither tried. The chemical structure of asphaltene is a multi-polymer system having variety of building blocks of flat sheet of condensed aromatic systems, interconnected by sulfide, ether, aliphatic, chains or naphthenic ring linkage. The condensed aromatic system of asphaltene also contains holes and gaps consist of heterocycle atoms coordinated to transition metal like vanadium, nickel etc. mostly generated by free radicals. Asphaltenes are complex system and thus need careful thermal and chemical evaluation to understand the possibility of converting them into applications like carbon fiber. It is important to mention here that direct conversion of asphaltene into carbon fiber is not possible, mainly due to its impurity, chemical composition inconsistency and low molecular weight and poor rheological properties. Therefore, two potential approaches can be possibly utilized to convert asphaltene into carbon fiber: (i) either convert asphaltene in mesophase pitch and meltspinning as precursor fiber, (ii) blending with PAN and wetspinning as precursor fiber to reduce the amount of PAN in the system. Hence, the key knowledge

generated in this project will be a method to convert Alberta oilsands asphaltenes into precursor fiber followed by converting them into carbon fiber by stabilization and carbonization. We assume that the chemical composition and aromatic content of Alberta oilsands asphaltenes provided to us by Alberta Innovates is close to commercially available asphaltene and there is no need for preliminary desalting or dewatering processes. The primary objectives of the project have been provided in Table 2 and kept consistent across the project:

Table 2 The project objective and related approach to achieve them

Objective	Approach
Preliminary analysis of as received asphaltene	As received samples will be characterized by using FTIR, TGA, DSC analysis to understand their thermochemical attributes
To remove the inconsistency, impurities and reducing the low molecularity in as received asphaltene	AOA samples will be chemically and thermally modified and subsequently characterized by TGA, DSC and FTIR to assess the effect of modification
Understand the feasibility of converting modified and unmodified asphaltene into carbon fiber	Trials with meltspinning and wetspinning to develop precursor fiber and analyze for their mechanical properties
Development of carbon fiber	The precursor fiber will be stabilized and carbonized at Deakin University facility
Characterization of developed fiber and develop a key learning plans for future scope	Quantitative and qualitative Characterization of developed carbon fiber for their mechanical and structural properties

Apparently, the entire project can be subdivided into three stages: modification of asphaltene, spinning trials for precursor fiber followed by stabilization, and carbonization and quantitative and qualitative analysis of the developed carbon fiber. Primarily, the modification of asphaltene is expected to narrow the molecular distribution, remove heterogeneity which is anticipated to further improve thermal stability. The improved thermal stability may support us in the meltspinning process. The thermal modification of AOA has been also expected to remove the possible impurities in the samples, especially related to sulfur, to observe its effect during the asphaltene processing. It can be manifested that the primary objective of the work is to develop carbon fiber either directly from asphaltene or modified asphaltene or in conjunction with PAN polymer.

PERFORMANCE MATRIX

Carbon fiber finds its application in the field of aerospace, automobile, sports, and marines etc. and primarily defined as the fiber containing 92% carbon in composition. They are available in short and continuous forms. In general, carbon fiber attains its high strength and modulus by the degree of crystalline alignment along the carbon fiber consisting of sp2 hybridized carbon atoms arranged two-dimensionally in a honeycomb structure. As stated earlier, carbon fibers are commercially produced from pitch and polyacrylonitrile. In general, carbon fiber are categorized as ultra-high modulus (≥500 GPa), High modulus (≥300 GPa, strength to modulus ratio 1%), intermediate modulus (≤300 GPa and strength-to-

modulus ratio above $1x10^{-2}$), low modulus type (modulus lower than 100GPa and low strength), High strength type (strength more than 3GPa). Therefore, the targeted application of carbon fiber is varied with the intended application. Since the market of the carbon fiber is mainly covered by polyacrylonitrile based precursor system, therefore, a number of alternative precursor materials have been investigated in the recent years, especially for their targeted application in light weight vehicles. To keep the consistency in the development, the US Department of Energy (DOE) has defined the expected properties of carbon fiber as tensile modulus of around 172GPa and Strength 1.7 GPa. Given the limited timeline of the project, the minimum criteria for the development of carbon fiber was deliberated to the DOE target and decided as a performance parameter for the success of developed carbon fiber in the current project. When compared to the mechanical properties of currently available carbon fiber, the projected value may be limited but with the given timeline and first ever trial, this could be a well-defined milestone to achieve.

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

MATERIALS

The Alberta oil sand samples were obtained from InnoTech Alberta (a subsidiary of Alberta Innovates), named as Asphaltene sample bank S2. The analytical properties of sample have revealed carbon as a major constituent, but sample also contained very high weight fraction of sulfur as impurities (7.1 %mass) apart from hydrogen, nitrogen, oxygen additionally with other metal impurities. The sample in the project is denoted as AOA and any sample named, as received or pristine stands for the same. A total of six different solvents and an ionic liquid were used throughout this work, but to protect patentability the identities of these solvents are not revealed in this report and instead referred to as “solvent 1”, “solvent 2”, “solvent 3”, “solvent 4”, “solvent 5”, “solvent 6”, and “ionic liquid”.

ANALYSIS AND MODIFICATION OF ALBERTA OIL SAND ASPHALTENE (AOA) SAMPLES

Analyzing and understanding the physical and chemical nature of asphaltene is key for its effective utilization as precursor material. AOA samples are not uniform in their chemical structure and contain various heteroatoms like nitrogen, sulfur, hydrogen, oxygen apart from metal constituents like nickel, vanadium, aluminum etc. which significantly changes the characteristics of asphaltene. Additionally, low aromaticity and wide distribution of molecular weight can hinder the practical application of asphaltene. Therefore, it is essential to modify as received AOA sample to narrow the molecular weight distribution, increase aromaticity and reduce the possible impurities. To modify asphaltenes samples, a number of methods have been proposed such as heat treatment, solvent extraction, hydrogenation, or catalytic modification. In this project, heat treatment and solvent extraction method have been selected to modify the AOA samples and subsequently analyze the resulted products.

Extraction is generally referred to as the separation of insoluble, low molecular weight fractions from any samples with the use of suitable solvents under particular ambient condition. Solvent extraction and ultrasound sonication methods are considered as the simplest, cheapest and easiest method compared to many other extraction methods like pulsed electric filed, super critical fluid extraction, negative pressure cavitation etc. Solvent extraction method primarily works on the principle of solubility of solute in respected solvents. As revealed from analytical study of Alberta sample, it contains insoluble asphaltene weight fraction of 89.7 wt% and 37.9 wt% in pentane and heptane respectively. Therefore, we have selected the dispersion of AOA samples in the mixture of solvent 1/solvent 2 and solvent 2/solvent 3 respectively to remove any soluble part and collect the insoluble part as the main product. This method is known as de-asphalting process. As reported earlier, there are number of methods that have been reported for the extraction process for asphaltene to remove low molecular weight fractions and soluble content. In the current project, we have exploited the possibility of conventional method of solvent extraction, ultrasound assisted extracted method in addition to thermal modifications. Thermal modification of samples has been carried out by utilizing general purpose furnace and tube furnace. Apart from these extraction methods, extraction from ionic liquid has been also utilized for modification of asphaltene. The obtained extracted samples were characterized by FTIR, DSC and TGA to contemplate chemical and thermal changes.

SPINNING METHODS AND EXPERIMENTAL PROCEDURE TO PREPARE PRECURSOR FIBER MELTSPINNING

The typical meltspinning set-up has been demonstrated in Figure 3 which consists of three zones: feed zone, melting zone and pumping zone. The initial zone of the system transports the feed material to the melting zone where it is heated to a temperature where material achieves spinnable viscosity. The spinnable viscosity of the pitch-based material largely depends on type of pitch and its mesophase content. The melting zone further transports the molten material to the pumping zone which contains a spinneret with multiple holes that exits oriented precursor fiber which later collects on a fiber winding device. Since the fiber is already drawn during the process of winding therefore, there is no requirement of additional drawing during the carbonization process. Meltspinning method is considered an effective method of producing carbon fiber precursor due to its high productivity and solvent free spinning. Although, spinning just received asphaltene or tar-based system (with no further treatment/modification) present a significant challenge and may not be even possible. Especially, spinning asphaltene requires preprocessing to transform it into either isotropic or mesophase pitch due to its poor rheological properties and low melt strength.

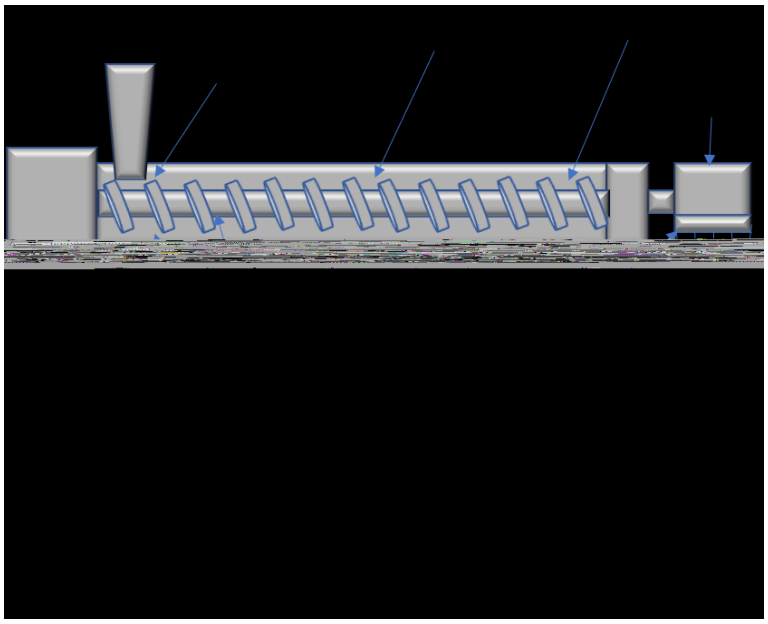


Figure 3 Schematic illustration of typical meltspinning set-up

In the current project we have utilized single screw extrusion for meltspinning AOA samples as shown in Figure 4 (a). It is important to acknowledge that melt-spinnable tar-based system must be a mixture of high molecular weight mesophase with few side groups. It is also important to note that the presence of high impurities and metallic content is not acceptable for meltspinning pitch material. To develop, carbon fiber from asphaltenes, it is important to transform asphaltene to isotropic pitch. Isotropic pitch is primarily used for spinning general purpose carbon fiber. In order to develop high strength fiber, it is important to convert isotropic pitch into mesophase pitch. In the current abstraction, we have not quantified the amount of isotropic and mesophase content in modified or heat-treated AOA samples due to lack of time. Although, the number of trials was conducted to understand the ability of as received AOA and chemical and thermally modified asphaltene for melt spinning. The meltspinning of as received samples was carried out in various temperature ranges. It is observed that pristine AOA samples were not able to form fiber at any of the temperatures and observed to have severe fumes mainly due to the degradation of the sample as time progresses. Similarly, thermally modified samples from tube furnace, general furnace were also tried for meltspinning. None of the samples were found to be spinnable. All the samples were either producing severe fumes or flowing without formation of any filament or fibers. To develop melt-spun fiber from AOA, thermoplastic polymers were also examined as shown in Figure 4 (a) and (b). We were able to develop fiber from asphaltene and polymer blend, but these trails were not further extended since it was outside the scope of the proposed work. It is postulated that developing fiber from AOA via melt spinning needs further detailed analysis as follows:

i. Possibility to convert mesophase from AOA via chemical and thermal methods
ii. Characterization of developed mesophase and its content
iii. Detailed understanding is required to understand the rheological properties with respect to temperature

Since, it was difficult to examine every aspect in the six-month time frame therefore, main focus was directed to the wetspinning of AOA samples, which is discussed in subsequent sections.

PRECURSOR FIBER SPINNING

In the current project, we have utilized the wetspinning set-up at Deakin university which has the capacity of producing 100 and 1000 filament tow as shown in Figure 8. As delineated in the image, wetspinning setup consists of five zones: spin pack (containing dope, filter, gear pimp and spinneret), coagulation bath, washing and drawing bath, spin finish, drying setup (how air drying column and heated godet) and finally winder for collecting precursor fiber. Dope for the spinning experiment was prepared with previously explained modified process.

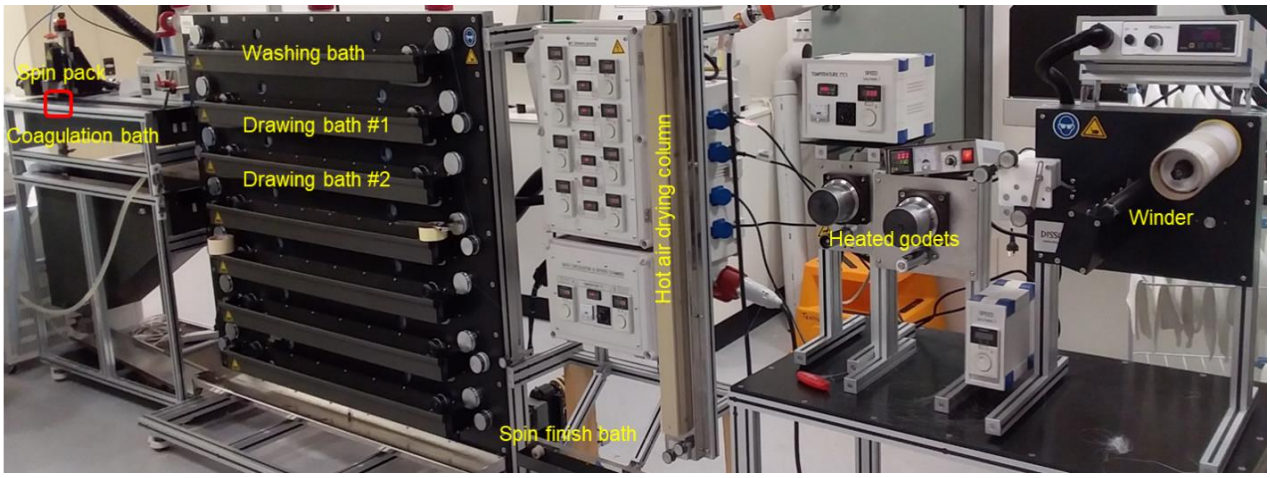


Figure 8 Wetspinning setup in Deakin University with the capacity of producing 100 and 1000 filament tow

STABILIZATION AND CARBONISATION

PAN precursor fibers have glass transition temperature of ~80℃ and exhibit melting temperature of about 350℃. However, PAN starts degrading prior to the initiation of melting. Therefore, conversion of PAN precursors to carbon fiber (CF) involves stabilization followed by carbonization. Essentially, oxidation is one of the most important processing stages for converting precursor fiber in high strength CF. The oxidation process is performed in controlled heating (180-300℃) condition under controlled tension and air environment to avoid melting, fusing, and burning of the fiber during high temperature carbonization process. Fiber oxidation process can be effectively carried out by isothermal heating, but this method is not practically possible in industrial setup. In general, stepwise heating and single step heating are another two methods to achieve fiber stabilization. However, stepwise heating is the industrially preferred method. Generally, oxidation process undergoes cyclization, dehydrogenation and oxidation that transform PAN structure to non-degradable and non-flammable ladder like structure. Carbonization process is mainly performed in the temperature range of 400℃ to 1500℃ in inert (N2) environment. Carbonization process reduces the weight of the fiber by about 50% through the loss of gases and byproducts like ammonia, hydrogen cyanide, carbon monoxide, carbon dioxide, nitrogen, hydrogen, and possibly methane. The carbonization process facilitates the formation of graphitic structure in lateral direction of fiber via coalescence of cyclized sections.

Carbon Nexus in Deakin University houses two fully functional production lines (research line and pilot scale production line) with the capacity of 3k to 320k tow sizes as shown in Figure 9 (a) and (b) respectively. The fully integrated research line is primarily focused on research and development of small tow size of

the precursor fiber. Since, the current project involves the feasibility study of developing carbon fiber from AOA asphaltene samples, therefore, we have utilized inhouse carbon fiber simulator for stabilization and carbonization as illustrated in Figure 9 (c). The line capacity of carbon fiber simulator was noted to be <3k filaments. As illustrated in Figure 9 (c), CF simulator contains separate stabilization and carbonization units. The oxidation process in CF simulator can be performed in stabilization unit in four different zones with four different temperature conditions. Similarly, the carbonization process was also performed in five different heating zones with varying resident time.

(a)

Creel

Oxidation Ovens

LT Furnace

HT Furnace

Surface Treatment, Sizing

Winding

(b)

Fibre In Feed

Oxidation Ovens

Material Handling

Carbonisation Furnaces

Surface Treatment, Sizing

Winding

Abatement

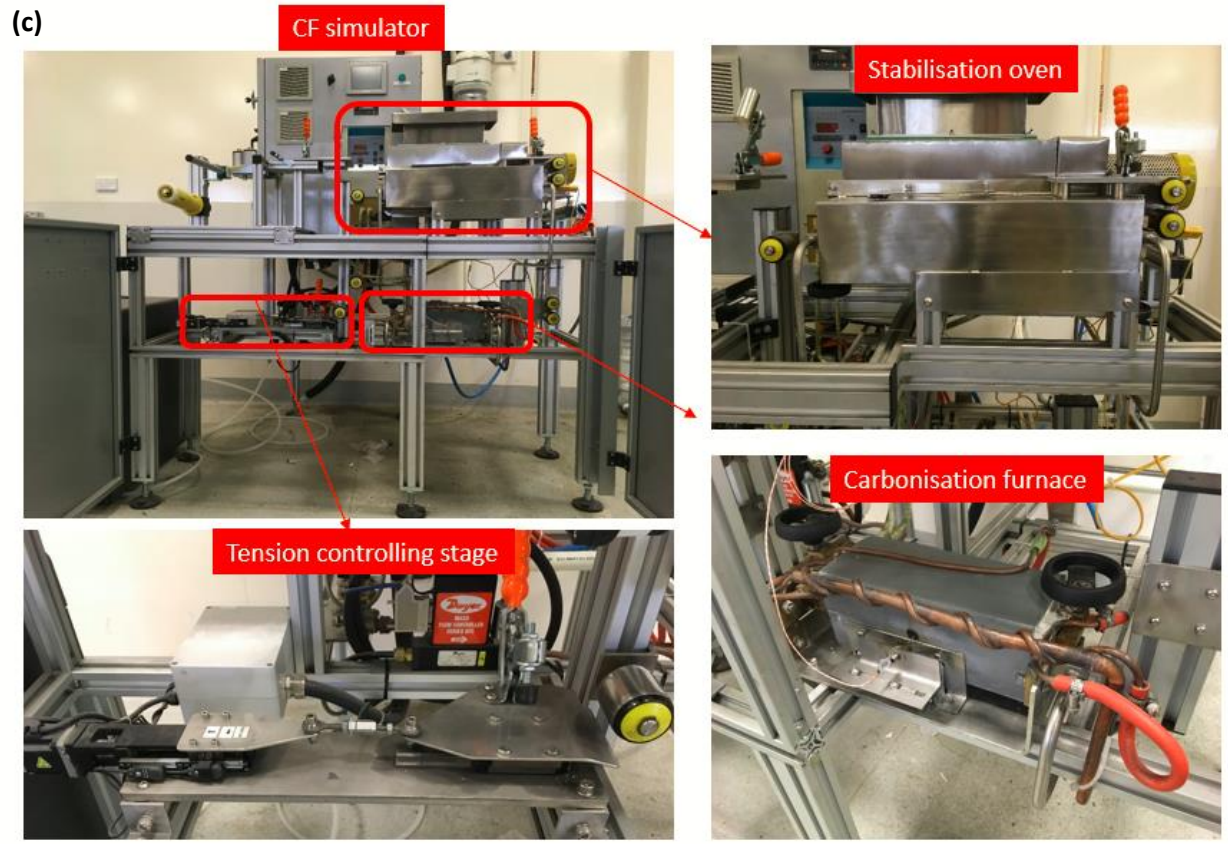


Figure 9(a) The single-tow carbon fiber research line at Carbon Nexus (b) The pilot scale carbon fiber line at Carbon Nexus (c) Inhouse carbon fiber simulator for stabilization and carbonization

CHARACTERIZATIONS

LINEAR DENSITY AND MECHANICAL PROPERTIES

An automatic single-fiber test system (FAVIMAT+ ((Textechno, Germany)) was used to measure the linear density and mechanical properties of fibers, under ambient room temperature and humidity (Figure 10). The mechanical properties of the fibers were tested at a crosshead speed of 10 mm/min for precursor and 2mm/min for carbon fiber, a gauge length of 20 mm, and with a 210 cN load cell. For each fiber type, 25 filaments were tested, and the average value was reported.

Figure 10 Favimat- single fiber tensile testing machine

RAMAN ANALYSIS

The change in disordered (D-band, sp3) and graphitic (G- band, sp2) structure of the carbonized fibers were analyzed using a Renishaw inVia Raman microscope (Figure 11). Three random spots on the fibres were picked to calculate the changes in the disordered to graphitic (I_D/I_G) ratios.

Figure 11 RAMAN analysis instrument

THERMAL GRAVIMETRIC ANALYSES (TGA)

The thermal stability behavior of the modified AOA samples was examined by a TA thermogravimetric analyzer (TA instrument, TGA Q50) (Figure 12).

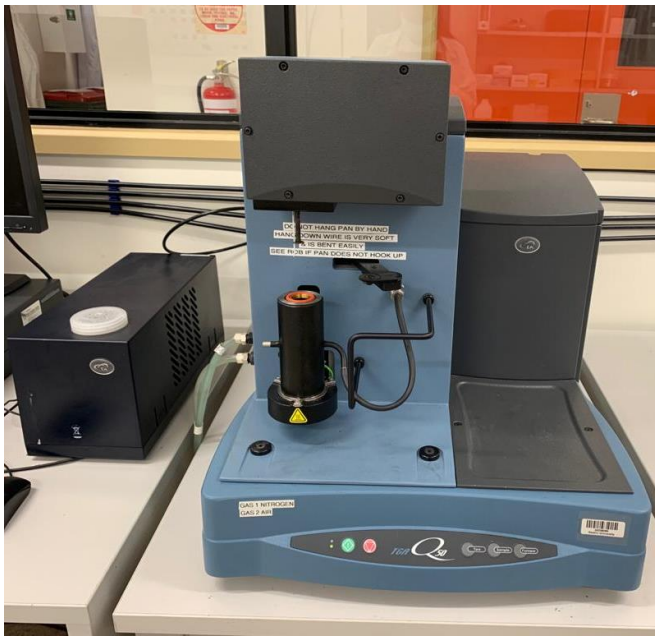


Figure 12 Setup for TGA analysis

DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Differential scanning calorimetry was performed using a TA instrument DSC Q200 to study the thermal properties of modified AOA (Figure 13).

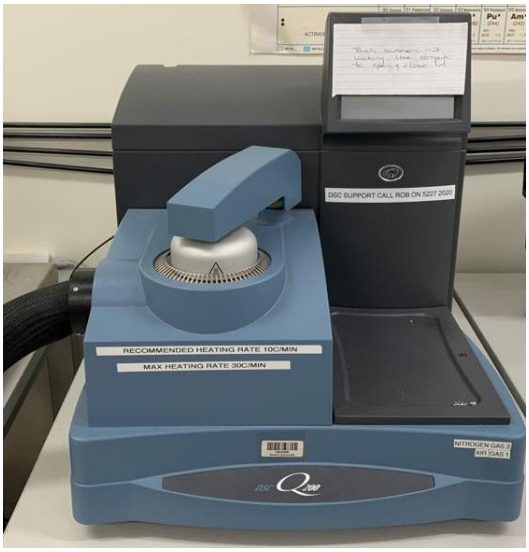


Figure 13 DSC analysis tool for modified samples

SCANNING ELECTRON MICROSCOPY (SEM)

The surface morphology of the fibers was analyzed by a scanning electron microscope (Zeiss Supra 55VP, Germany) (Figure 14).

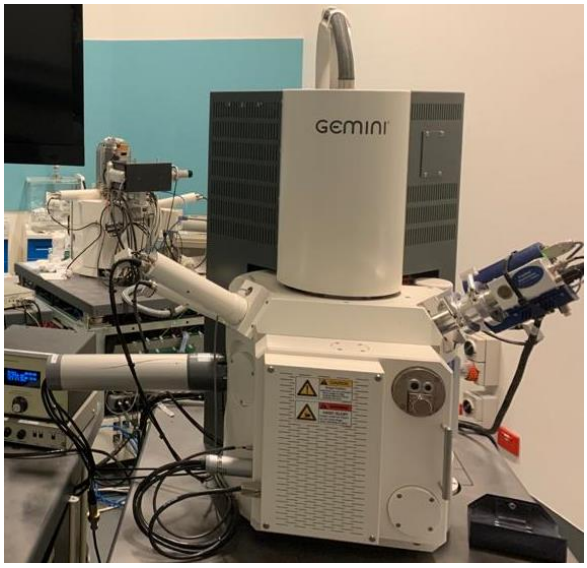


Figure 14 Scanning electron microscope to observe the morphology of the fibers

FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

FTIR spectra of the stabilized were measured using a microscope FTIR (Bruker LUMOS FTIR) Figure 15

Figure 15 FTIR instrument for analyzing EOR value for the stabilized fiber

D. 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.

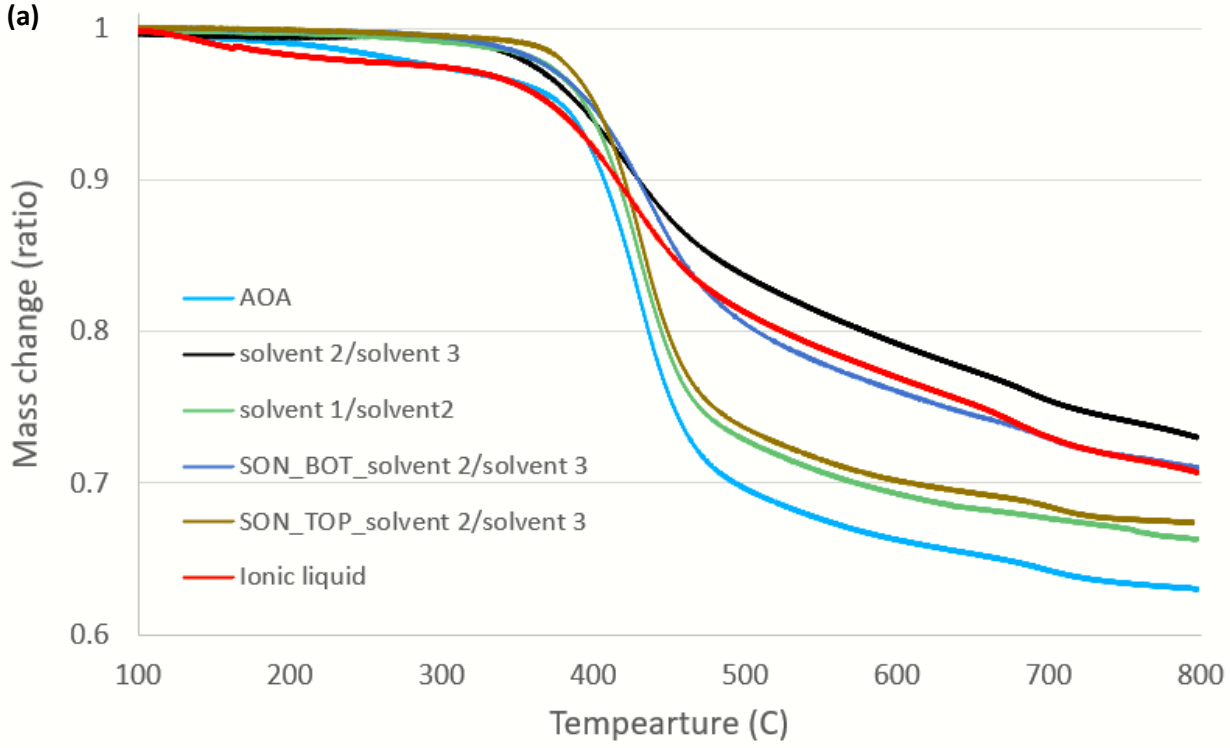
RESPOND BELOW

MODIFICATION OF AOA SAMPLES

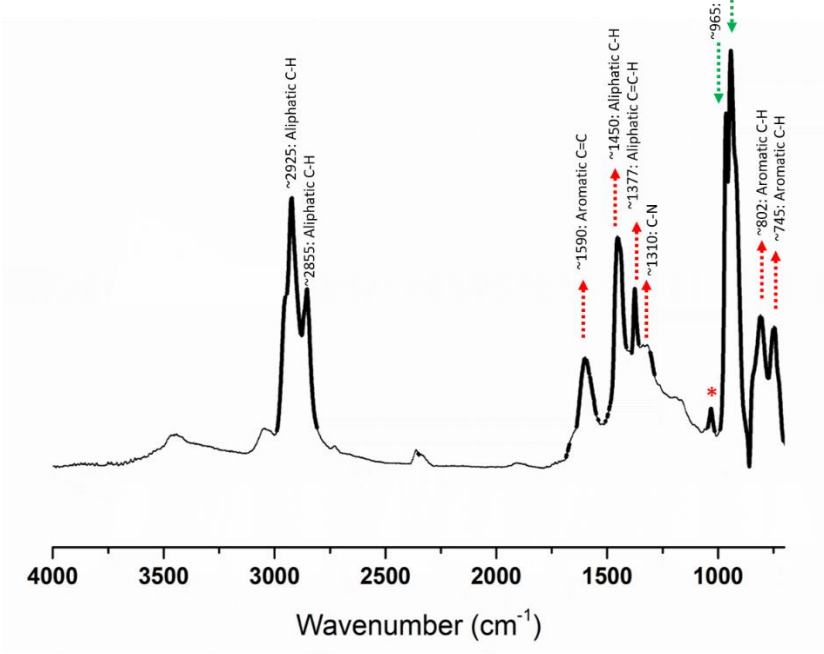
SOLVENT EXTRACTED AOA (CONVENTIONAL AND UAE)

Solvent extracted samples were first examined by the TGA to understand the change in their thermal behavior or weight loss with respect to the temperature and modification methods (conventional extracted method and UAE extracted method). Figure 16 (a)illustrates the TGA analysis modified samples which clearly demonstrates that all the modified samples have shown improvement in thermal stability compared to as received AOA samples. Interestingly, as received samples and ionic liquid modified samples have different slope after the initiation of decomposition process which clearly indicates the possibility of faster rate of degradation process for these two samples. As stated in the previous section, the AOA samples exhibited the tendency of phase separation during the ultrasound assisted extraction process. The TGA analysis of top and bottom residue have not indicated much distinction in their thermal attributes. The maximum weight loss of both the samples were observed in the temperature range of 350℃-480℃. The similar behavior was observed for as received AOA samples too. Among all the extraction process, the conventional method of extracting asphaltene from the combination of solvent 2 and solvent 3 was found to be the most effective pathway. Solvent 2/solvent 3 samples were thermally more stable and did not exhibit severe weight loss for any temperature ranges. The detailed FTIR analysis of as received samples have revealed that AOA have notable aliphatic, aromatic and polyaromatics other than sulfoxide group as shown in Figure 16 (b). The available structural details have been provided in FTIR image of the AOA samples. When compared to the as received FTIR, the extracted samples did not show any notable change in the structure either conventional extraction or ultrasound assisted extraction process as shown in Figure 16 (c). It is clearly evident that the change in thermal stability of the extracted samples especially solvent 2/solvent 3 is not related to the change in chemical structure. In fact, thermal stability was presumably achieved by the removal of volatile content which was not traceable in FTIR. To evaluate the effect of solvent extraction on AOA, further evaluation was conducted by DSC.

(a)


Mass change (ratio)
1
0.9
0.8
0.7
0.6
100
200
300
400
500
600
700
800
Tempearture (C)
AOA
solvent 2/solvent 3
solvent 1/solvent2
SON_BOT_solvent 2/solvent 3
SON_TOP_solvent 2/solvent 3
Ionic liquid

(b)


~2925: Aliphatic C-H
~2855: Aliphatic C-H
~1590: Aromatic C=C
~1450: Aliphatic C-H
~1377: Aliphatic C=C-H
~1310: C-N
~965: C=C Alkenes
~942: C=C Alkenes
~802: Aromatic C-H
~745: Aromatic C-H
*
4000
3500
3000
2500
2000
1500
1000
Wavenumber (cm-1)

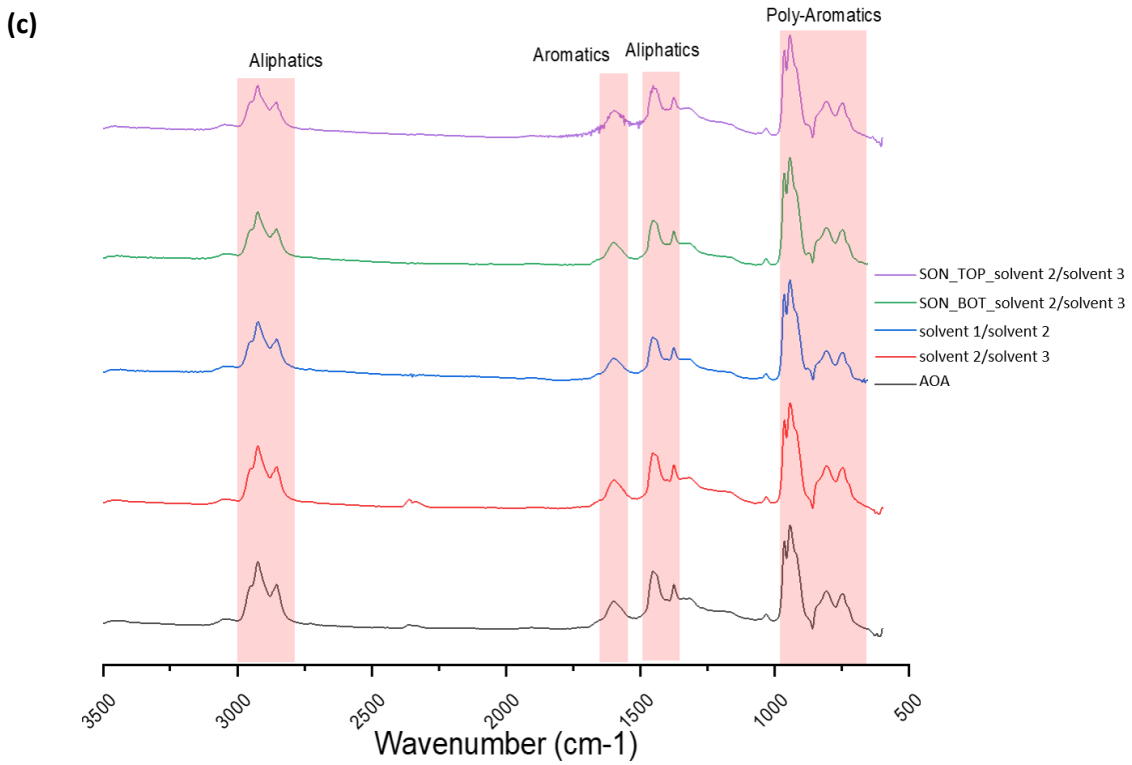


Figure 16(a) TGA analysis of chemically and UA extracted samples (b) FTIR analysis of as received AOA (c) comparison of change in structure after chemical and UA extracted sample

The obtained DSC thermograph for all samples were noticed to be the same except solvent 1/solvent 2 and SON_BOT_solvent 2/solvent 3 samples as shown in Figure 17. As evident from TGA analysis, weight loss was observed for all samples, but it was prominent for as received AOA and solvent 1/solvent 2, which was similarly found in DSC analysis. It is also important to mention here that many studies have been carried out to identify the glass transition temperature of various asphaltene. The obtained data for glass transition temperature has been noted to have very broad range (~100-300℃) which indicates the compositional change variation in asphaltenes. Additionally, the variation in the glass transition temperature of asphaltene is also related to the measuring technique, heating rate, thermal and physical pretreatment of samples. However, such endothermic peaks for evaluating Tg for all extracted samples was not found to be discernable or values were creating ambiguity in the results, therefore, further analysis on Tg was not extended. In future, detailed understanding of Tg can be a prominent tool to contemplate the change in physiochemical behavior when AOA is solvent extracted or modified with any other methods.

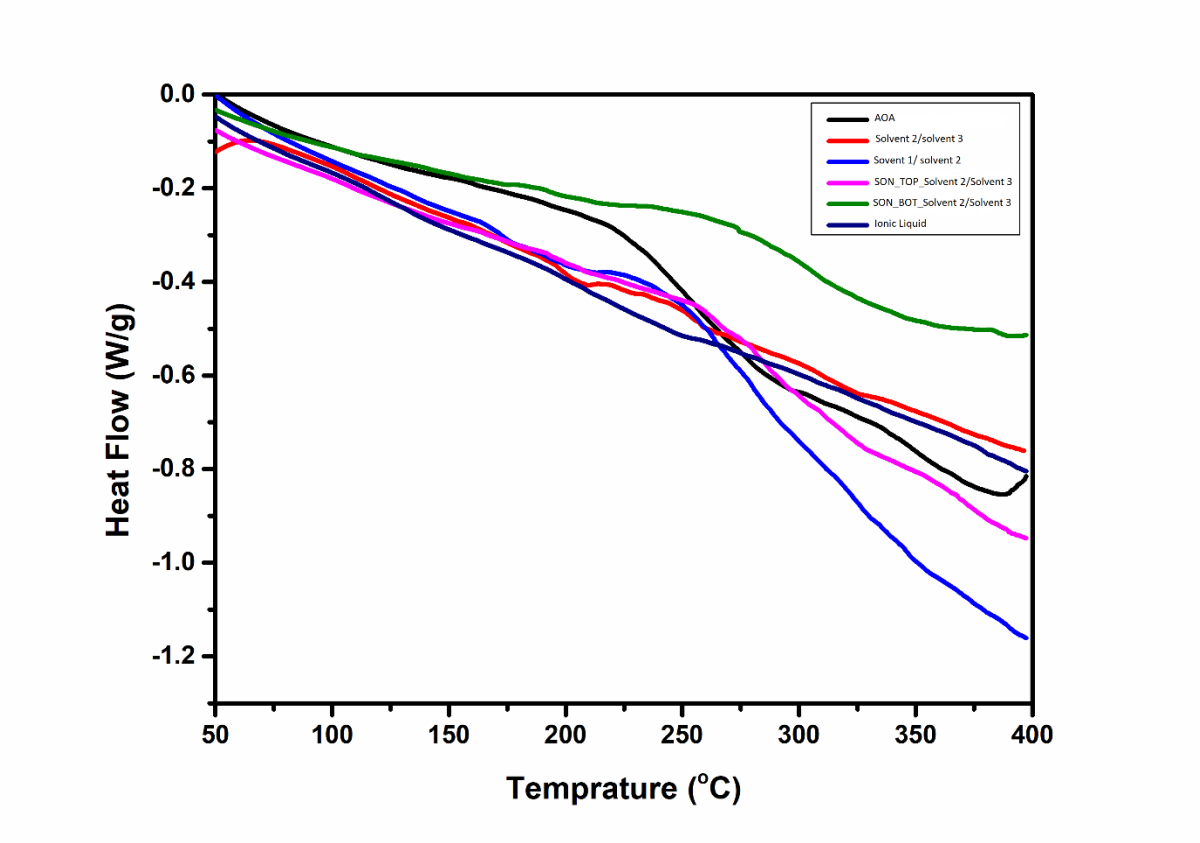


Figure 17 DSC analysis of the modified samples

THERMAL MODIFICATION

The initial study of thermal treatment of AOA samples have shown significant change in chemical structure of the AOA. Apparently, heat treatment method was found to be more effective than solvent extraction method. Apart from change in chemical structure, heat treatment also causes the removal of sulfur when asphaltene samples were heated in the range of 50 to 600℃. However, heat treatment is not a method which fully removes the sulfur content from AOA. As evident from Figure 18 (a), increasing heat treatment temperature increase the aromatic content of AOA simultaneously reducing the sulfur content. This phenomenon was limited to the higher temperature. It was observed that at higher temperature, all the aliphatic and aromatic content was found to be eliminated which could be corroborated to the degradation of AOA. To ensure the postulation of thermal degradation we have separately conducted heat treatment at higher temperature for varying resident time. The resulting samples were further examined by TGA analysis as elucidated in Figure 18 (b). As delineated in Figure 18 (b) the TGA spectrum of heat-treated samples for longer resident time is found to have severe weight loss followed by full degradation at higher temperature.

To further compare the effect of heat treatment process on AOA, all the heat-treated samples were examined for DSC and obtained result has been shown in Figure 18 (c). Similar to the solvent extracted samples, DSC thermogram for heat-treated samples were observed to be endothermic without any distinguishable peak for calculating glass transition temperature. However, number of important key finding were observed in heat treatment process of AOA, detailed as follows:

I. AOA samples started degrading when the temperature is increased beyond a certain limit, which indicated the most critical thermal behavior of AOA.

II. There are number of desulfurization techniques like catalytic, dehydrogenation, thermal and processing through ionic liquid but thermal processing is one of the easy and effective methods. However, it cannot fully remove the sulfur content from AOA. The heat treatment process has clearly demonstrated its ability for removing sulfur from as received AOA. But it will be interesting to understand low sulfur affect mechanical properties of developed carbon fiber.

III. Apart from removing sulfur, heat treatment process was not found to be effective to assist meltspinning. Heat treated samples did not form fibers which needs to be analyzed in detail. Since the time frame of the project is limited therefore, much focused was given on wetspinning process which has shown significant potential for developing carbon fiber.

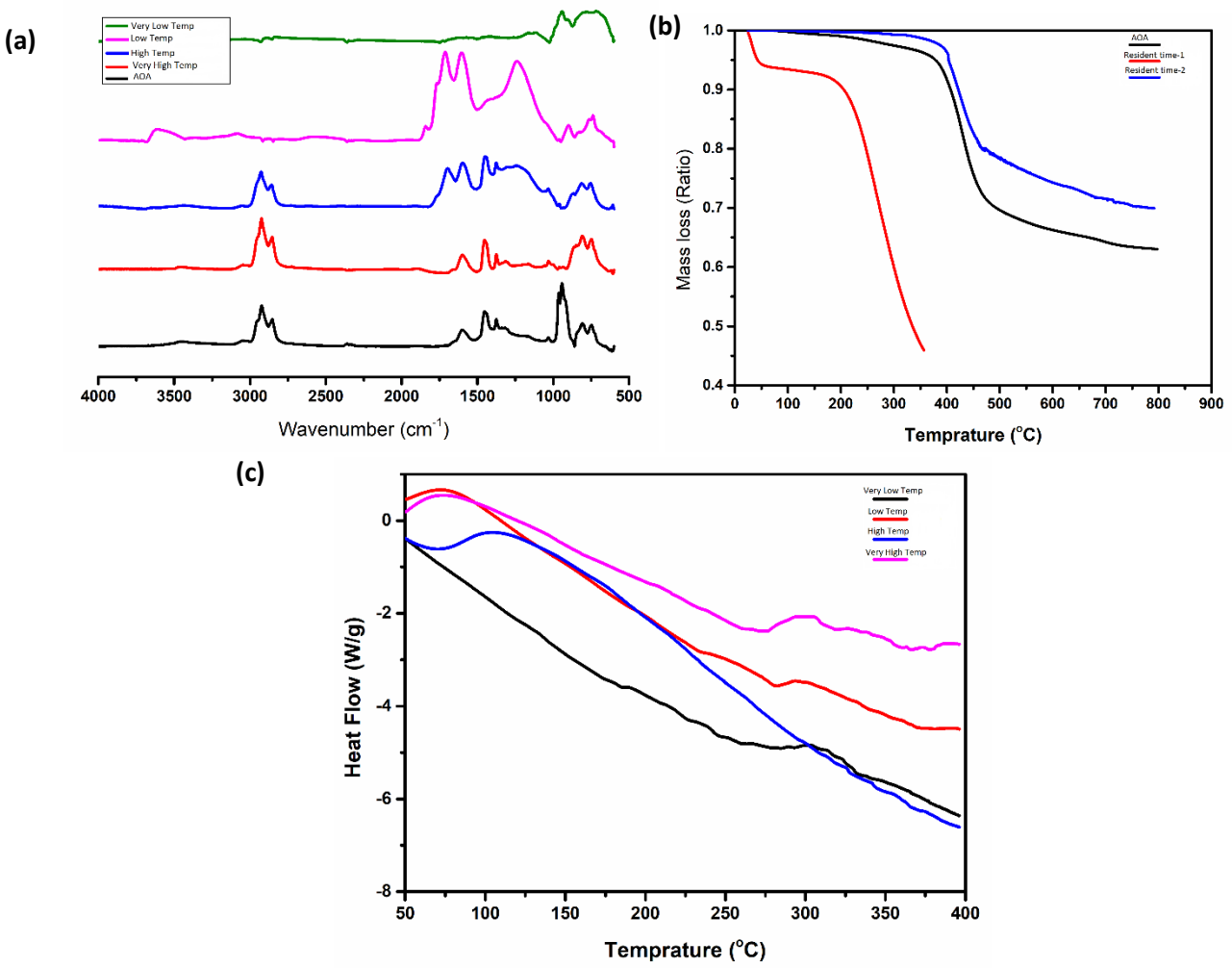


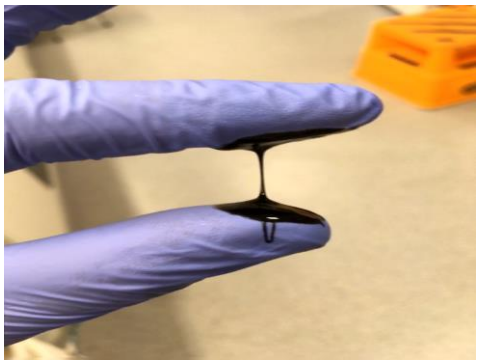
Figure 18 Analysis of AOA samples when heat treated in the temperature range of 50℃ to 600℃ (a) FTIR analysis of heat treated samples at various temperature (b) TGA analysis of heat treated samples at higher temperature for varying resident time (c) DSC of heat treated samples

WETSPINNING OF PRECURSOR FIBERS

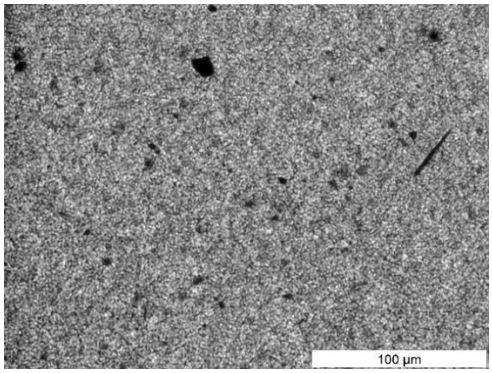
The compositional detail of wetspinning process has been provided in the previous section of spinning methods and experiments. The major decision at this point was the selection of type of AOA for wetspinning process. At the start of wetspinning process, we had four different type of AOA i.e. as received, solvent extracted, ultrasound extracted and thermally modified. The decision was made on isothermal analysis of as received AOA samples. The isothermal analysis of as received sample have demonstrated less than 1% of mass loss at given temperature and time. Therefore, it is possible to include As-Received AOA in wet-spinning dope for preparing precursor fibers and stabilize fibers without major damage.

The methods of dope preparation have been already elaborated in the previous section. To ensure the preparation of spinnable dope we have conducted two fundamental analysis on dope including physical visualization of dope and microscopic study. In a very simple term, the spinnable dope needs to be able to sufficient strong to sustain its own weight when physically stretched as shown in Figure 19(a). It was also observed that if the dope contains larger agglomerates in solution, it created unexpected conditions like severe blockage to the spinneret, reduced fiber plasticity, limited drawing, and poor mechanical properties. It was observed that when agglomerates size is more than permissible limit, it was not spinnable due to blockage at the spinneret holes. The spinnable size of agglomerates was largely dependent on filter mesh which is coupled with the spinneret to filter any possible physical impurities to be suspended in fiber. All the compositions were microscopically examined as shown in Figure 19 (b) to (d). The physical and microscopic evaluation of dope has provided the fundamental understanding over the dope preparation method. The homogenously developed dope has rendered successful spinning of various wt% of AOA/PAN precursor fiber as shown in Figure 19(e).

(a)



(b)



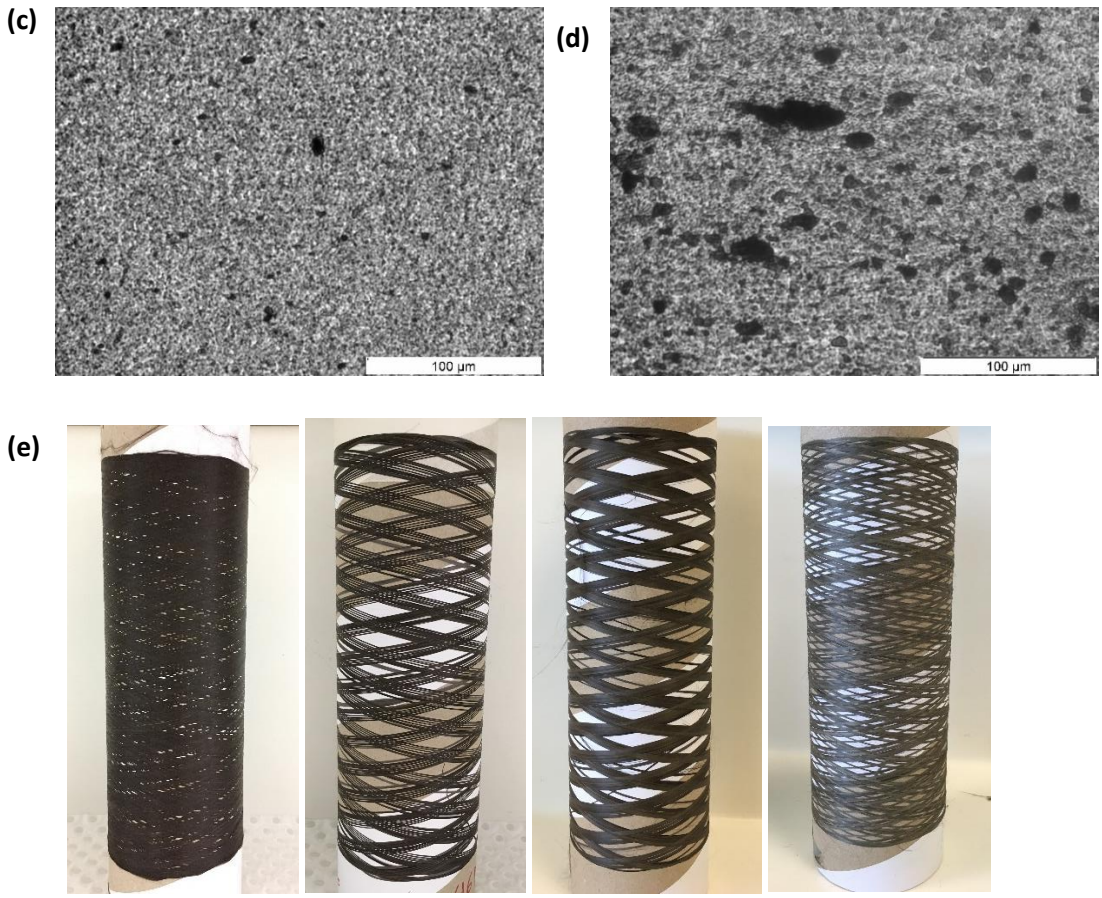


Figure 19 physical testing of spinnable dope (b) microscopic analysis of very low wt% AOA samples (c) microscopic analysis of low wt% AOA samples (d) microscopic analysis of high wt% AOA samples (e) Wet spun PAN/AOA precursor fiber with varying weight fraction of AOA

MECHANICAL PROPERTIES OF PRECURSOR FIBER

The PAN based precursor fiber generally demonstrates the typical tensile strength of ~500MPa, tensile modulus of ~11GPa, elongation of around ~11-12% and diameter in the range of ~11-13µ. However, these mechanical properties are too general and highly dependent on PAN precursor type and their suppliers. To obtain mechanically acceptable precursor fiber, there are number of parameters like coagulation bath temperature, extrusion rate, drawing ratio, drying bath condition, drying temperature, winding speed etc. which need to be optimized during wetspinning process. The initial few trials were not successful because the acceptable precursor properties especially diameter was not obtained during the spinning.

To evaluate the mechanical properties of developed precursor fiber, single fiber filament test was carried out by Favimat. 25 fibers were selected randomly to find the statistically correct results. The mechanical properties of all the composition was found to be significantly varying and found to be very low compared to the pristine PAN fiber, especially elongation in the precursor fiber. It was evident from results that increasing the weight fraction of AOA has further reduced the mechanical properties and mechanical properties were no more acceptable for precursor fiber. Force and elongation curve for all the compositions were found to be very scattered which indicates that not all the fibers are equally elongated during the drawing process as shown in Figure 20. To improve the mechanical properties of wetspun fiber,

number of modifications have been carried out in the spinning process in terms of drawing, temperature of coagulation bath and washing bath etc. It is well established that precursor fiber with higher fiber diameter and lower elongation do not provide carbon fiber with good mechanical properties. In order to improve the fiber elongation and scattering of force and elongation curve, post modification of all the precursor fibers have been carried out. The post modification method was believed to reduces the dipolar interaction between nitrile group that facilitates the precursor fiber stretching by virtue of plasticization. The fiber stretching imparts orientational angle of molecular chains with respect to the fiber axis.

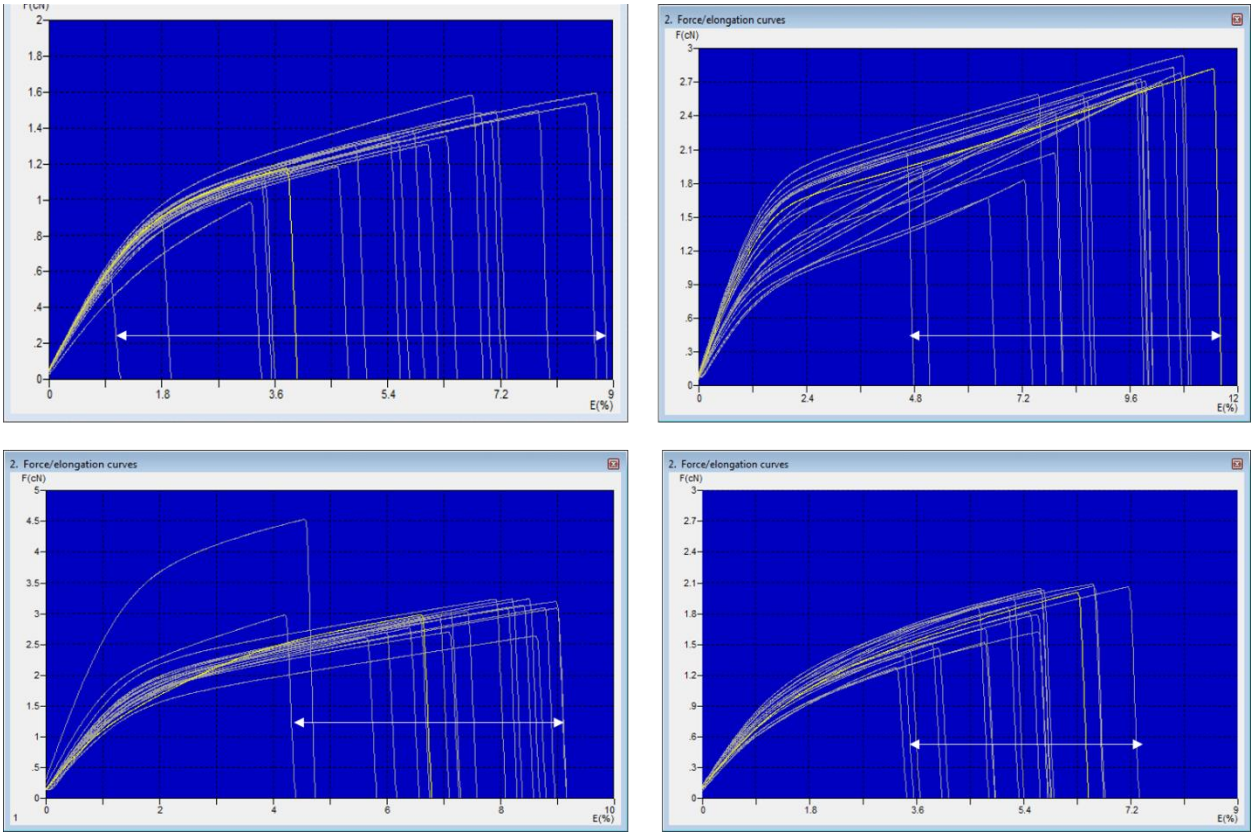


Figure 20Force vs elongation curve for varying wt % of AOA, indicating insufficiently elongated fiber in tow

FTIR SPECTROSCOPY AND EXTENT OF REACTION

The precursor fiber stabilization is one of the most crucial processing stages for producing carbon fiber. The stabilization process is generally performed in air at temperature range of 180-300°C. The tension force is also applied during stabilization process to prevent the fiber shrinkage or producing elongation to the fiber. It is observed that when PAN fiber is thermally treated in such condition it undergoes approximately 25% of shrinkage due to the formation of nitrile conjugation cross-links between the polymer chains. The stabilization process converts thermoplastic PAN into cyclic non-plastic cyclic compound that possess ability to sustain in high temperature environment during carbonization process. The stabilization process is mainly measured by the extent of reaction (EOR), obtained from FTIR analysis. EOR value is generally expressed in terms of % and calculated based on eq 1. In general, the preferred value for PAN based stabilized fiber should be in the range of 60 to 70%.

$$E = \frac{0.29I_{1595}}{0.29I_{1595}+I_{2243}} \quad \text{.....Equation 1}$$

The initial trials were conducted in general fan forced furnace (Figure 21) and once the condition was finalized then entire experiment was extended to carbon fiber simulator. The resulted data has been presented in Figure 22.

Figure 21Fan forced high temperature furnace

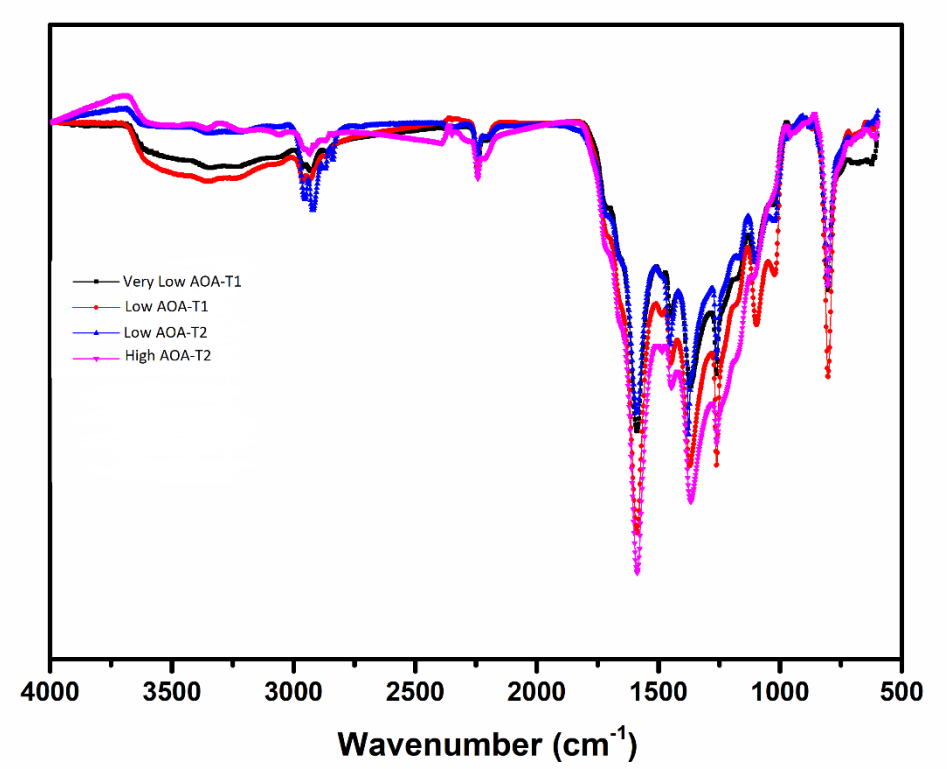


Figure 22 FTIR analysis and representation of EOR value for stabilized fiber

DEVELOPMENT OF CARBON FIBER

After stabilization, Carbon fiber is produced by pyrolyzing the stabilized fiber at the temperature range of 400-1500℃ in inert environment (commonly N_2). Carbonization process facilitates 50% weight reduction in fiber due the evolution of gases like ammonia, carbon mono oxide, water etc. The carbon simulator comprises an induction-based heating system which is very efficient to maintain temperature but cannot be run for longer time at high temperature. The limited capacity for resident time may not be suitable for larger tow but it was very effective for smaller tow size (400-500 filament). The varying

resident in the carbonization process is referred as trial 1 (T1) and trial 2 (T2). For higher weight fraction of AOA, high resident time was found to be deteriorating the fiber stability and continuous fiber breakage was noticed. The obtained mechanical properties for developed carbon fiber have been provided in Table 3.

Table 3 Mechanical properties of Carbonized AOA fibers

Composition	Elongation (%)	Diameter (µm)	Strength (MPa)	Modulus (GPa)
Very Low AOA-T1	0.4	7.34	515	155.62
Very Low AOA-T2	0.45	7.26	711	168.12
Low AOA-T1	0.28	6.89	330	128.56
Low AOA-T2	0.52	7.86	780	154.56
High AOA-T1	0.34	7.3	400	127.39

To further study the structure of the developed fiber, we have exploited the RAMAN spectroscopy to identify the G (graphitic) and D (disordered induced) bands in carbon fiber. When, ladder structure of the PAN fiber coalesces to form the graphitic structure along the axis of carbon fiber, it generates some form of disordered turbostatic structure (sp3 carbon atom) instead of fully ordered graphitic structure (sp2 carbon atom). Therefore, the ratio of D-band and G-band is manifested as qualitative parameter for carbon fiber. In general, the relative intensity of D- band (I_D) and G-band (I_G) are widely influenced by the number of factors including size of the sp2 graphitic structure, ratio of sp2/sp3 hybridization ratio, disordered bond etc. Figure 23 elucidates the RAMAN spectrum of developed AOA carbon fibers. The RAMAN results have revealed that carbon fiber with better mechanical properties have lower I_D/ I_G ratio i.e. the carbon fiber which exhibit the higher mechanical properties possess lower disordered structure along the axis of the fiber. At this, it is difficult to postulate that AOA contributes in improving the graphitic structure. However, pitch-based carbon fiber is known for their high modulus. Thus, it can be envisioned that the high modulus (comparable to DOE target of 170 GPa) of the carbon fiber in the current project may be contributed from AOA. This hypothesis needs to be evaluated in detail later.

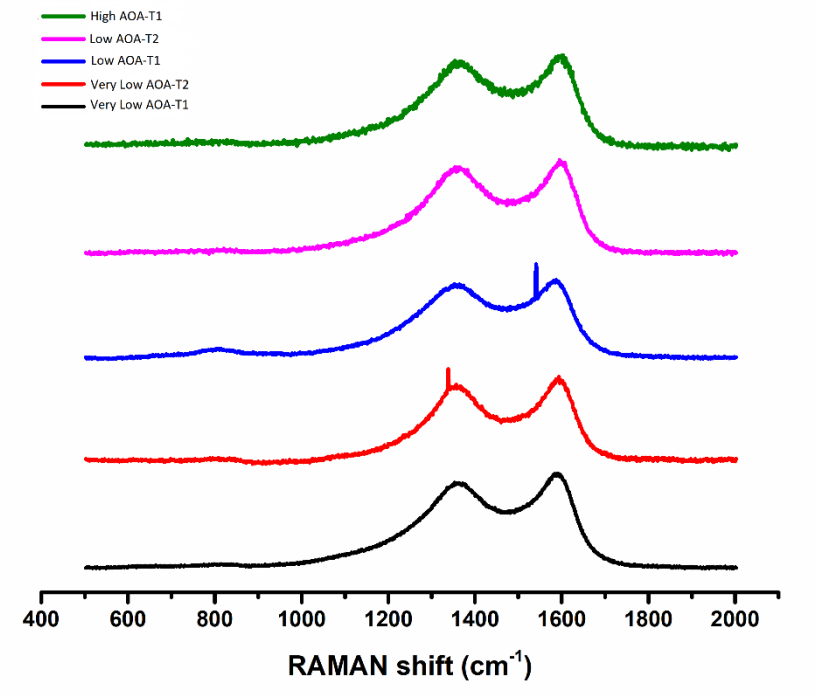
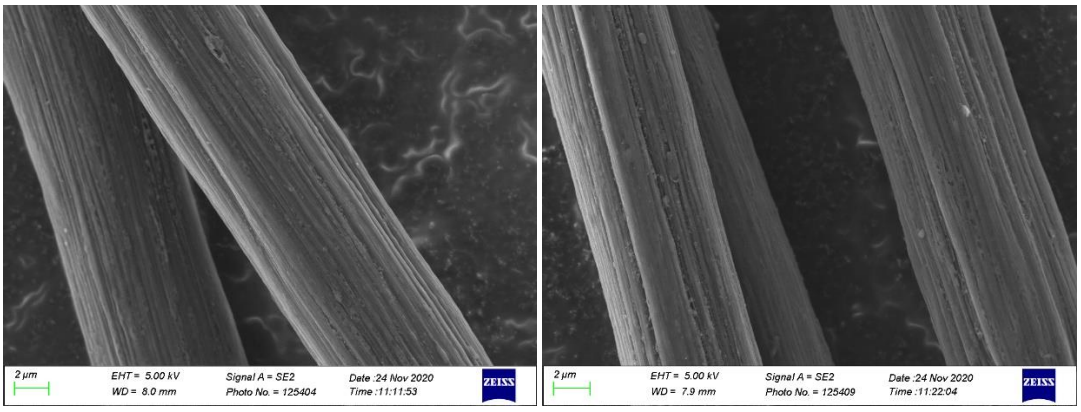


Figure 23 RAMAN analysis of developed carbon fiber

Thermal gravimetric analysis of just received AOA has shown the degradation phenomenon at high temperature. Therefore, we were expecting that the carbonization process may impact the surface morphology of the fiber due to the release or loss of AOA. It was interesting to note that the developed carbon fiber is mostly free from any of the defects like voids etc. Grooves on the surface of the carbon fibers are typical textural appearance of PAN-based carbon fiber as delineated in Figure 24. Based on our prior experience on working with PAN fiber, it can be clearly indicated that AOA derived carbon fiber possess huge potential.



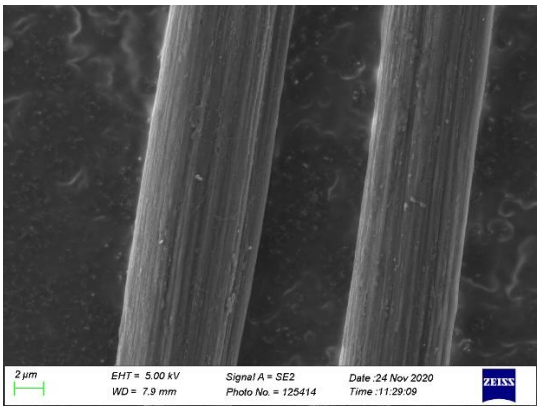


Figure 24 SEM images of various wt% of carbon fiber

PROJECT METRICS

Project Success Metrics (Metrics to be identified by Applicant)			
Metric	Project Target	Commercialization / Implementation Target	Comments (as needed)
Tensile strength	*172MPa*	*3000 MPa*	*We anticipate exceeding the DOE (Department of Energy) minimum requirements and should achieve a higher tensile strength*
Tensile modulus	*172GPa*	*250 GPa*	*We anticipate exceeding the DOE (Department of Energy) minimum requirements and should achieve a higher modulus*

The project achieved the tensile strength metrics and fell just short of the modulus metric.

E. KEY LEARNINGS

Please provide a narrative that discusses the key learnings from the project.

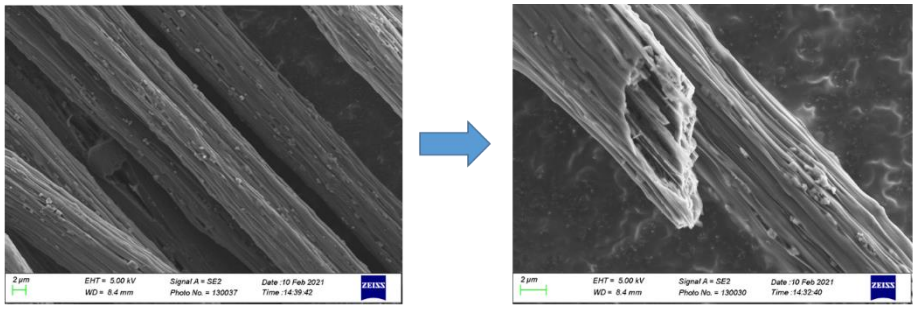
- Describe the project learnings and importance of those learnings within the project scope. Use milestones as headings, if appropriate.
- Discuss the broader impacts of the learnings to the industry and beyond; this may include changes to regulations, policies, and approval and permitting processes

RESPOND BELOW

There are number of observations noted in the project including the importance of homogenous dope preparation, and selection of solvent with minimum impurities to prepare the asphaltene based dope for wetspinning process. It has been observed that the presence of a high fraction of impurities in the co-solvent system causes phase separation that contains an asphaltene rich phase and a polymer rich phase as shown in Figure 25. To circumvent the formation of phase separation, a number of iterations have been carried out to identify the actual cause. Additionally, the formation of homogeneous dope was also found to be very important in developing carbon fiber with acceptable mechanical properties. As evident from Figure 26, if dope contains agglomerated, non-dispersed and non-homogeneous solution, it causes the development of severe defects on carbon fiber surface. As stated earlier, asphaltene utilized in this project undergoes degradation at higher temperature. Therefore, it is possible that the hollow cross-section

observed by the microscopic analysis is due the severe degradation of asphaltene during thermal treatment.

Figure 25 Precipitation observed in dope preparation



After modification of dope preparation method

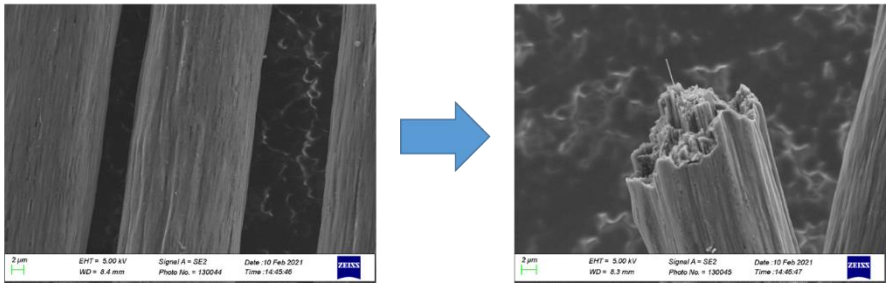


Figure 26 Illustration of importance of dope preparation along the axis of the carbon fiber

F. OUTCOME 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

Carbon fiber is generally manufactured from polyacrylonitrile but its exploitation to the full potential is limited due to variable and high cost. Therefore, alternative precursor materials are actively investigated all around the world. However, the replacement for PAN based carbon fiber is not yet realized commercially. The current project is focused on to understand the feasibility of converting Alberta oil sand asphaltene as a sustainable alternative to current carbon fiber precursor. The focus of the project was to develop carbon fiber with the mechanical properties close to the DOE target from Alberta oil sand asphaltene either by meltspinning or wetspinning process. The project outcome has demonstrated significant potential in terms of developing carbon fiber especially from wetspinning process. Apparently, the initial study has also shown that current asphaltene can also be meltspun if its investigation is further extended. The preliminary results in this study have shown the potential for patent to secure the knowledge generated in the current work. Although, the current work is limited to the small-scale laboratory trails with limited timeframe. But it is envisioned that this project has potential to manufacture carbon fiber at commercial level if project is further extended at the larger scale trials.

CLEAN RESOURCES METRICS

Clean Resources Metrics (Select the appropriate metrics from the drop down list)			
Metric	Project Target	Commercialization / Implementation Target	Comments (as needed)
$ Future Investment		10,000,000	Anticipate significant capital to be required for commercialization .
# of Publications	1	5	At least 1 publication from Phase 1 project
# Students (Msc., PhD, Postdoc)	1	10	Budgeted for 1 CI (in-kind) and one post doc for 6 months Phase 1
# Patents filed	1	5	Anticipate one patent arising from success in this Phase of the Grand Challenge.
Partnership agreements / MOUs?	1	2	Will look to partner with suitable carbon fibre manufacturer
# New products/services created	1	2	New precursor derived from Alberta Oilsands
# New Spin-Off Companies created	1	1	Dependant upon outcome of Phase 1
$ in Clean Technology	$50,000		Alberta Innovates investment

The current project is envisioned to develop the process and methods for converting Alberta oil sand asphaltene into carbon fiber. The first phase of the project is to contemplate the feasibility of converting asphaltene into carbon fiber in the duration of 6 month. As mentioned in the clean resource metrics, team is working towards the completion of 1 publication in the form of review article on conversion of asphaltene into value added applications like carbon fiber and other carbonaceous materials for applications like energy storage, as reinforcement in polymers etc. Further discussion is also extended to convert the obtained results in this project into technical publication. However, the technical publication depends on terms of confidentiality of the work. One postdoc worked on the project and a patent may be filed in the near future based on the work. Further development may potentially lead to partnership agreements/MOUs, new products/services being created, and new spin-off companies being created.

PROGRAM METRICS

Program Specific Metrics (Select the appropriate program metrics from the drop down list)			
Metric	Project Target	Commercialization / Implementation Target	Comments (as needed)
# of End Users participating	Phase 1 will be conducted at Carbon Nexus. Phase 2 of this this project will have a composites manufacturer	For commmercial implementation we will require multiple end users to validate the performance of the fibres	Deakin Univesity has an extensive network of Carbon Fibre industry partners who can assist in commercialisation if required.
Unique product/process	1	1	
# commercial BBC products	0	1	

A unique process was developed in this project meeting the project target listed above.

G. 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

The global market for carbon fiber was expected to grow at US$3.8 billion in the year of 2020 bit further revised to grow at US$7.2 billion with the compounded annual growth rate of 9.5% in the analysis period of 2020-2027. The market of carbon fiber is mostly covered by the aerospace and defense sector. It is well known that 90% of the carbon fiber is developed from polyacrylonitrile (PAN) but the utilization of PAN based CF is limited due to the high cost. Therefore, developing alternative precursor material can be one of the most conscious development in this field. It is clear that employing the knowledge developed in this project may mitigate the dependency on PAN and its varying cost. Producing CF from Alberta asphaltene can fulfill the economic mandate by accomplishing the following:

i. The developed CF may potentially provide new commercial opportunities not only in the field of automotive but it can be also utilized in other applications like marine vessels, flame proof structure, medical applications, acoustics, replacement for fiber glass in the composites. The range of CF application demonstrates its ability to attract many sectors and subsequently cater the development of new opportunities and jobs.

ii. Asphaltene is a byproduct in petroleum field and readily present in Canadian oil fields. It is mainly contributing to the high viscosity of the oil. The development of CF can be one of the most value-added application of asphaltene. The development of low-cost CF with acceptable properties from asphaltene will pave the way for new avenues and industries to produce new class of material based on asphaltene CF.

iii. Asphaltene related issues causes costs billions of dollars per year and even causes production reduction, low production capacity due to the well shut in. Therefore, it is valuable to recover the asphaltene management cost by utilizing asphaltenes for developing asphaltene-based commercial products.

ENVIRONMENTAL

Canada accounts for 1.6% of total greenhouse gas emission in the world as per CAIT, 2017 and 26% of this total emission is contributed by oil and gas industry. Although, data on the environmental implication of asphaltene has not been much available in public domain but commercial use of asphalt have significant environmental impact. When asphaltene is separated from oil sand then its industrial utilization of may cater the philosophy of sustainable growth and avoided carbon dioxide emissions from combustion.

SOCIAL

The Alberta government in 2009 led a plan, called as "*Responsible Actions: A Plan for Alberta's Oil Sands"* which aimed to provide development by balancing environment, social and economy. The economic development was proposed to achieved by utilizing six major strategies: developing Alberta oil sand in an environmentally responsible way, promoting healthy communities and quality of life, maximize long term value by economic growth stability and resource optimization, proactive approach to Aboriginal consultation, maximizing resource and innovation to support sustainable growth and enhancing the accountability in the management of oil sands. This project is envisioned to strengthen the idea of idea of sustainable development and economic growth by resource optimization. The success of the project ensures the possibility of growing local business and developing an intellectual property which can be utilized at global platform. The idea of this project will contribute to the enhancement in quality of life and communities in the form entrepreneurship opportunities.

BUILDING INNOVATION CAPACITY

Although the project was conducted in Australia the learnings can ultimately be implemented in Alberta and lead to attraction of HQSP from outside the province to Alberta.

H. 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 primary focus of this project was to understand the Alberta oil sand asphaltene and its ability to transform into carbon fiber. The application of carbon fiber is wide from low scale application to high end aerospace application. Therefore, any change or modification in this filed have capability to reach and alter the to the wide range of industries and organizations. But the information available for commercially available carbon fiber is mostly intellectually protected or classified. The fundamental knowledge in this field is available in the form of either journal publication, public reports, book chapters etc. In this context, the knowledge developed in the current project will be disseminated in the form of public report and journal publication. It is important to mention here that the current work involves the utilization of specific process parameters, modification methods and techniques which hold the potential for developing patent to protect the process for the development of carbon fiber from AOA samples.

I. FUTURE SCOPE AND CONCLUSIONS

Carbon fiber is finding its prominent presence in number of applications around the globe and its potential is not completely exploited due to the high production cost. It is known that the precursor cost is the main contributing factor in production of carbon fiber. A number of alternative precursor materials have been sought for developing low cost carbon fiber. Therefore, two important strategies have been employed to find a low cost carbon fiber alternative (i) develop a new alternative like lignin, rayon etc. to counter the higher cost of precursor material (ii) reducing the PAN precursor weight fraction by replacing the it with other fillers or blend. In this project, the possibility of utilizing Alberta oil sand asphaltene as an alternative to the currently available precursors was investigated. The finding in this work has contributed to the development of new concepts and strategies for finding a new alternative in carbon fiber feedstock. This project is unique and Alberta oil sand asphaltene for the first time were utilized as precursor for carbon fiber. When comparing the mechanical properties of currently obtained carbon fiber with other commercially and non-commercially available fibers, we may be far from some of the carbon fiber, but very close to the targeted tensile modulus of DOE (for low-cost carbon fiber). However, tensile strength is still low compared to DOE target. We envisioned that DOE target can be achieved with further optimization in spinning, stabilization, and carbonization of fiber.

i. The obtain properties of carbon fiber is satisfactory but we still need to evaluate the fiber in more details. The future scope in the project may also involve the evaluation of crystallographic orientation of the fiber since it is most important attribute of carbon fiber.
ii. In the current work, all the process was carried out on 100 filament tow size, but we experienced that when increasing the tow size exceptionally improves the mechanical properties. The effect of process optimization is found to be very prominent on the fiber when the tow size is large (≥ 1000 filaments). Therefore, the development of larger tow size should be considered in future aspect of the project.
iii. Apart from, considering the development of carbon fiber it is essential to undertake the cost analysis in the current project. Cost analysis will provide the clearer image of how use of AOA may contribute to the final cost of the carbon fiber derived from AOA.

In summary, based on our findings Alberta asphaltene holds promises for the development of low-cost carbon fiber, but more understanding of its processing and ways to improve its conversion to carbon fiber as well as effect of impurities in particular its sulfur content are required.