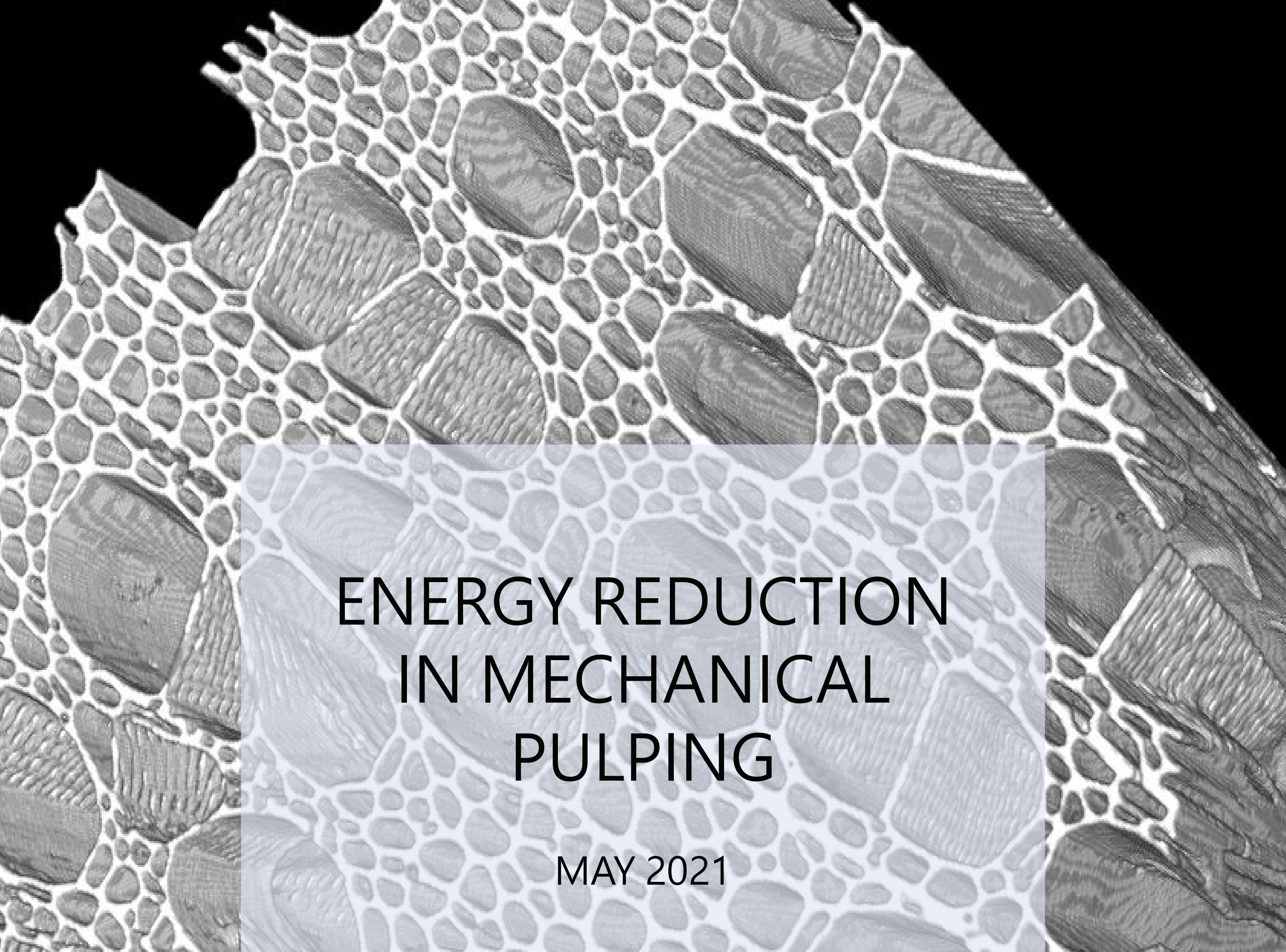

ENERGY REDUCTION IN MECHANICAL PULPING

MAY 2021

THE UNIVERSITY OF BRITISH COLUMBIA

WELCOME MESSAGE

Dear partners in the Energy Reduction in Mechanical Pulping research program,

Greetings from Vancouver. I am pleased to update you on the latest developments in the program. Since our last newsletter, we held a successful online Steering Committee meeting where students and the principal investigators presented project updates and introduced the proposed work for this new phase. This meeting was our first one using an online format, and we had a total of 65 attendees. We thank you again for your support and the valuable feedback provided to the ERMP team.

In good news for our team, we are happy to welcome Professor Emily Cranston back from her maternity leave. The ERMP team wishes you and and your new born all the best. On other exciting news, Bryan Bohn submitted his master thesis and successfully defended his M.A.Sc early this year. Matthias Aigner continues completing his research work and is currently working towards graduation. I invite you to read more about the team's progress and future work on the following pages. As we continue navigating through covid-19, our next Steering Committee meeting will be held online again on June 3rd through the Zoom platform. I hope you all have marked your calendars and will join us for this online event.

As the year progresses, we have also had several personnel updates with new highly qualified personnel joining the consortium. Postdoctoral fellows: Samira Gharehkhani, Mengqi Fang, Jingqian Chen, and Rasmita Sahoo have joined the program and are working closely with ERMP researchers to continue making progress towards project milestones. With this, we have completed the recruiting of all postdoctoral fellows for each research project. We have also welcomed new UBC graduate students, Ph.D. student Mariana Frias de Albuquerque, and M.A.Sc student Siwei Chen. A new staff member, James Drummond, laboratory microscopist, and wood imaging expert, is also supporting the ongoing research at UBC-PPC through imaging processing and analysis for several ERMP projects. I invite you to review pages 30 and 31 for brief introductions to our new team members and their backgrounds.

Wishing you the best and good health, hoping we can meet in person soon.

Sincerely,

Mark Martinez

Professor of Chemical and Biological Engineering, UBC
Principal Investigator, ERMP Research Program
Director of Advanced Papermaking Initiative, API

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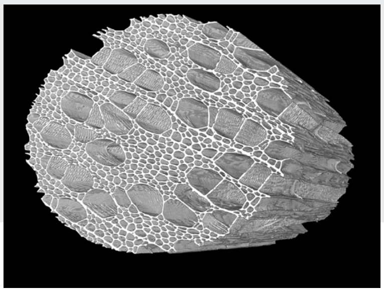

CONTENTS

RESEARCH UPDATES

PROGRAM UPDATES

CONTACTS

ON THE COVER

X-ray tomograph image of an untreated aspen wood chip using the Ziess Xradia 520 Versa at the UBC - PPC. Photo Credit: Aurélien Sibellas.

PROJECT 1.1

LC REFINER BAR FORCE BASED CONTROL STRATEGIES

Authors: Matthias Aigner, Samira Gharehkhani, James Olson, Peter Wild

Background

In previous work by researchers at the University of Victoria, a custom piezo-ceramic force sensor was developed to measure local shear and normal forces applied to the refiner bars during refining. This sensor, shown in Figure 1, b.), has a probe that replaces a short length of a refiner bar and that is sensitive to forces that are: (1) normal to the axial facing surface of the refiner bar and (2) normal to the long axis of the refiner bar and in the plane of the axial facing surface of the refiner bar Figure 1, a.). These forces are referred to as normal force (i.e.) and shear force (i.e.), respectively. Sensors based on this design have been used in trials in a variety of high consistency (HC) [1] and low consistency (LC) refiners [2], [3].

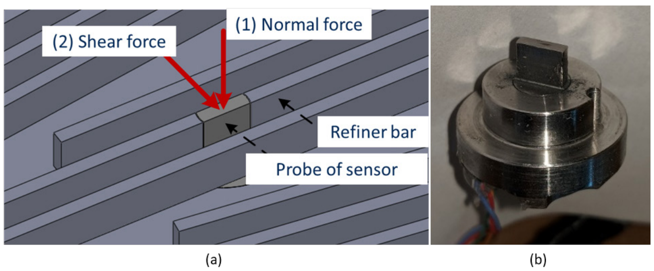

Figure 1. Force sensor set up a.) the direction of the measurable forces. b.) one of the bar force sensors.

In the first phase of the current project, this sensor was installed in the stator plate of the AIKAWA 16” single disk pilot LC refiner at the Pulp and Paper Centre at the University of British Columbia. This sensor was paired with a rotary encoder to investigate changes in bar-force profiles with respect to the relative position of the rotor to the stator. This work showed a strong correlation between the onset of fiber cutting and a distinct change in the measured bar-force profiles, namely the transition from a one peak force event to a two peak event per bar crossing event.

In the second phase of this project, six sensors were installed in an Andritz TwinFlo 52” LC-tertiary refiner at the Catalyst, Paper Excellence mill in Crofton BC (Figure 2). The goal of this trial is to investigate: side-to-side balance in the TwinFlo refiner; the magnitude, and radial distribution of forces and the effect of operating conditions on these forces; and, as in the work at UBC, the profile of forces during bar passing events. Three sensors are situated in each stator plate at different radial positions. In Figure 3 a.), positions of the sensors are indicated on a CAD drawing of a refiner plate, and in Figure 3 b.), the back of the stator plate is photographed, showing the housing of the sensor to protect the sensors against the environment inside the refiner.

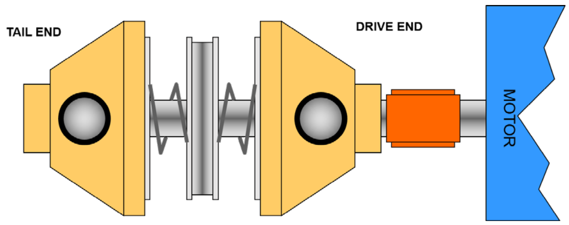

Figure 2. Layout of TwinFlo refiner at Crofton.

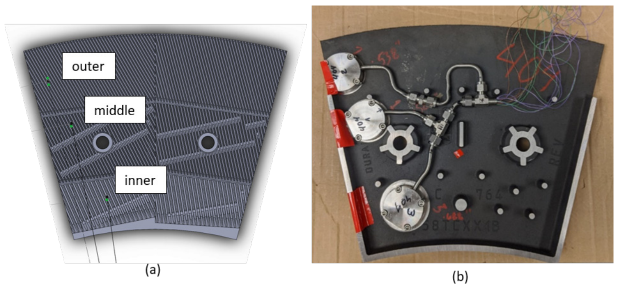

Figure 3. a.) CAD drawing of Stator plate. Sensor positions are indicated. b.) Picture of the stator plate back. Housing of sensors and wiring visible.

Commissioning of the refiner was on January 8th 2020. Since installation, the sensors have been recording forces during day-to-day operation. In addition, trials were conducted to collect force data as the refiner load is increased (i.e. power curves) and as the balance of discharge flow or discharge pressure between the sides of the refiner is varied (i.e. bias trials).

Force profiles during bar-passing events

In Figure 4, Figure 5 and Figure 6, the force profiles during a bar passing event are shown for the inner, middle and outer sensors, respectively, at the tail end of the refiner for SRE values ranging from 130 to 40 kWh/At. The duration of the bar passing event point is, on average, 0.37, 0.28 and 0.25 ms for the inner, mid and outer position, respectively. SRE set points where chosen in increments of 10 kWh/At, and a no load (NL) position was also included.

PROJECT 1.1

In the plot for the inner sensor (Figure 4), the peak force occurs close to the center of the bar passing event. This bar-force profile is consistent for all SRE values during this power curve.

For the sensor located in the middle of the stator plate (Figure 5) a transition in the force profile is noticeable. For SRE values below 70 kWh/At, only one force peak is visible and is located before the midpoint of the bar passing event. For SRE values above 70 kWh/At, two distinct peaks are present in the force profile, one occurring in the first half of the bar passing event and one in the second half of the event. Furthermore, the force rises more rapidly at the start of the bar passing event compared to the inner sensor.

For the outer sensor (Figure 6), a single force peak occurs approximately midway between the start and the midpoint of the bar passing event.

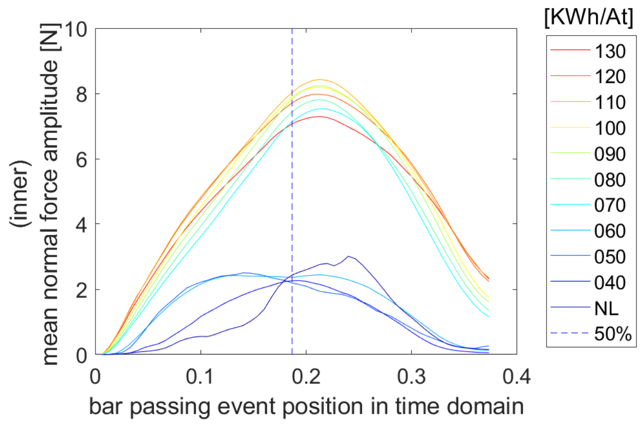

Figure 4. Force profile plot for tail end inner sensor for different specific refining energies. Bar passing event duration: 0.37ms. March 3rd.

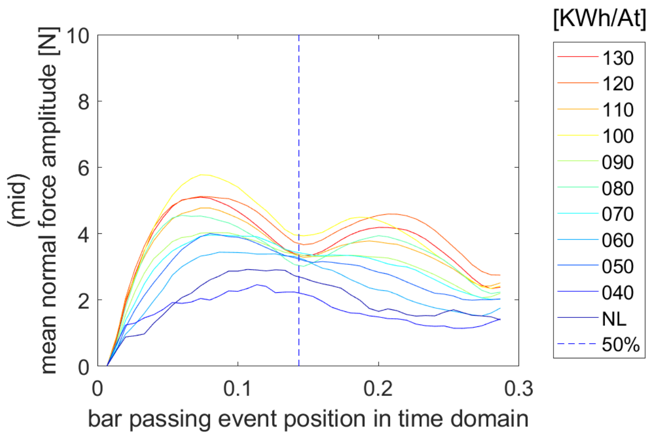

Figure 5. Force profile plot for tail end mid sensor for different specific refining energies. Bar passing event duration: 0.28ms. March 3rd.

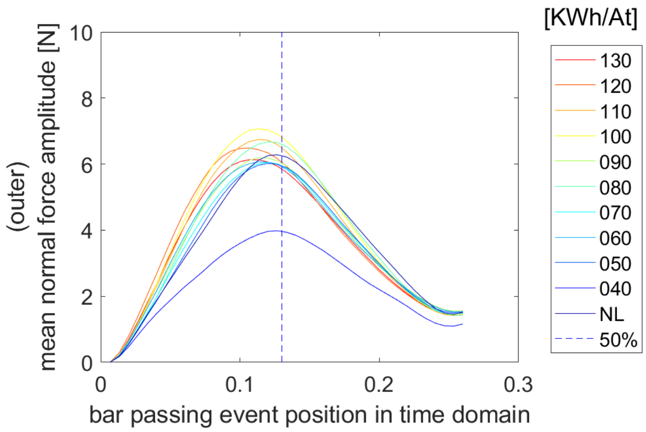

Figure 6. Force profile plot for tail end outer sensor for different specific refining energies. Bar passing event duration: 0.26ms. March

Previous work by our group, in the test refiner at the PPC-UBC, shows a transition from, a single force peak during a bar-passing event to two peaks, and that this transition corresponds to the onset of fiber cutting [4]. This result suggests that, based on the mean force profiles for the three sensors in the Crofton refiner, fiber cutting occurs predominantly in the middle region of that refiner, for the conditions tested.

The normalized fiber length data for the presented data shows a fiber length decrease of about 5% at the SRE value of 70 kWh/At where the transition is noticeable at the mid sensor (Figure 7). This correlates with findings from the UBC refiner where this transition to fiber cutting was identified by a fiber length decrease of approximately 7%.

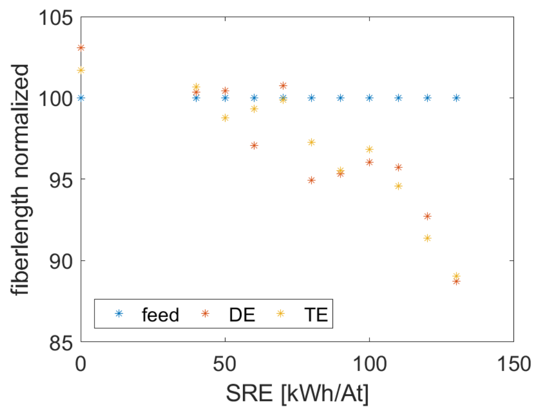

Figure 7. Normalized fiber length data for feed drive end (DE) and tail end (TE) discharge line.

PROJECT 1.1

The trials in the UBC refiner also showed that, where there are two peaks in the force profile, the peak in the later half of the bar passing event can be associated with friction force, and the peak in the earlier half can be associated with corner force. Additionally, the earlier peak (i.e. corner force) is only present in the fiber cutting range whereas the later peak (i.e. friction force) is present through the entire range of the trial. These results lead to several tentative interpretations of the Crofton data: First, the single peak at the inner sensor is due to a friction force and significant fiber cutting does not occur at this position. Second, the two peaks at the middle sensor represent a corner force and friction force. Third, for the outer sensor, the single peak in the force profile suggests that fiber cutting is not occurring. However, this force peak is skewed to the first half of the bar passing event, which, paradoxically, suggests fibre cutting in the absence of a significant friction force.

Future work

Further trails at the Crofton mill are planned, including an investigation of the balance between the two refiner sides. In these trials, a pressure bias is artificially introduced between the two refiner sides. Force measurements may shed light on refiner behaviour in a balanced and unbalanced states. Additionally, arrangements are being made to run further trials in the test refiner at the PPC-UBC with pulp from the Crofton mill. The purpose of these trials is to compare the force measurements taken at Crofton to measurements taken at the test refiner under similar refining conditions. The trials at UBC will be run at conditions that mimic the conditions at the radial location of the three sensors. These tests combined with the trial results from Crofton promise to improve the understanding of bar-force profile shapes.

Furthermore, fundamental and experimental studies will be conducted, to investigate the effect of plate pattern and pulp furnish on bar forces and the onset of fiber cutting. We will look at the bar force estimation model proposed by Kerekes and Meltzer [5] to find out to what extent it is valid for novel plate designs. Moreover, we aim to set the force level and SRE level where fiber shortening does not exceed the target level. The results will be compared to prior findings [5]. In addition, Kerekes and Meltzer reported that the target level of fiber shortening may be met at higher forces, but they did not have enough outputs to specify the conditions when this may happen. It is notable that, this trend has not been observed in our experiments, but future mill trails at Crofton and also novel plate designs might provide an opportunity to extend our knowledge in this regard.

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PROJECT 1.2 (A)

DATA ANALYTICS IN MECHANICAL PULPING PROCESS FOR REFINING PROCESS OPTIMIZATION AND MONITORING

Authors: Bhushan Gopaluni, Yankai Cao, Mengqi Fang.

Background

Motivation

Mechanical pulping processes are widely used to produce newsprints and other products with specific printing grades. Compared with chemical pulping processes, mechanical pulping processes produce much higher yield [1]. However, these processes are energy intensive due to the electricity consumed by the refiners [2]. A wide range of factors including variations in feed chip quality and unknown process disturbances drive the process towards non-optimal operating conditions leading to excess energy consumption. Our goal is to minimize electricity consumption while maintaining the pulp quality. In particular, we focus on a Chemi-Thermomechanical Pulping (CTMP) process is under research.

Harnessing the large volumes of historical process data, a series of data-based methodologies will be developed to analyze and assess the current approach to process operations, identify the potential opportunities for optimization, and build models to assist in process operations. Through data-based analysis, we will extract the latent process information from available historical measurements and prior process knowledge. Furthermore, the developed models will be implemented and adapted online.

Challenges

The CTMP processes exhibit certain characteristic features such as non-linearities, and stochastic disturbances making process optimization and monitoring challenging. These challenges include:

(1) The operating conditions for a specific type of CTMP process vary from site to site, and therefore the opportunities for energy reduction need to be appropriately customized.

(2) The CTMP process is high dimensional, and has correlated process variables. These correlations among the variables need to be accounted for in a systematic way to build models.

(3) The CTMP contains multiple lines and units. Some of them, such as refiners, directly consume energy and other units, such as chip pre-processing or latency removal units [1], affect the energy consumption indirectly. There exist several opportunities for energy optimization in this process, however, due to complicated correlations among process variables, the energy optimization strategy needs to be designed by taking a wholistic approach on the entire refining process.

Proposed problems and methodologies

In the next few months, we hope to tackle the following problems:

(1) Offline historical data analysis

Historical process data often have significant information about the current operating practices and their impact on the overall energy consumption and performance. We will therefore start with a thorough analysis of the historical data. Figure 1 shows our proposed workflow for offline analysis of process operations. This workflow includes: (1) data collection, data pre-processing, data synchronization and cleaning (2) separation and ranking of historical data into desirable and undesirable operating regions. This analysis allows us to identify operating regions with low/high energy consumption that satisfy the pulp quality constraints.

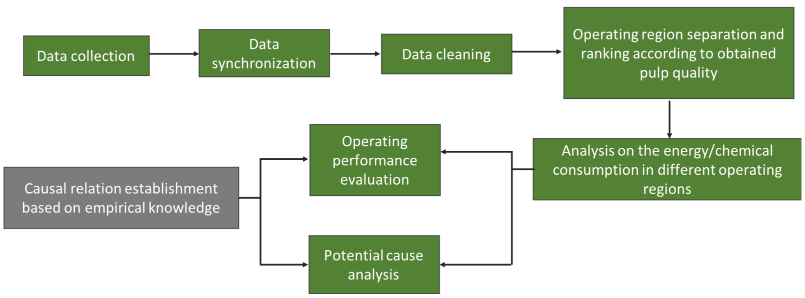

Figure 1. A general workflow of offline historical data analysis.

(2) CTMP refining process optimization

Figure 2 illustrates a schematic of a two-stage CTMP process [3]. Four potential future research directions for optimization are elaborated below:

PROJECT 1.2 (A)

(a) Optimization of energy allocation between the two-stages in high consistency (HC) refining process

The primary refiner in the 1st stage refining mainly processes wood chips and it has a smaller impact on reducing the freeness than the secondary refiner, which processes pulp directly. Using historical data for a given total specific energy, the specific energy consumed by primary and secondary refiners will be correlated with the resulting pulp quality. Our goal is to find the best energy allocation for desired pulp quality using models for the two-stage HC refining [4,5].

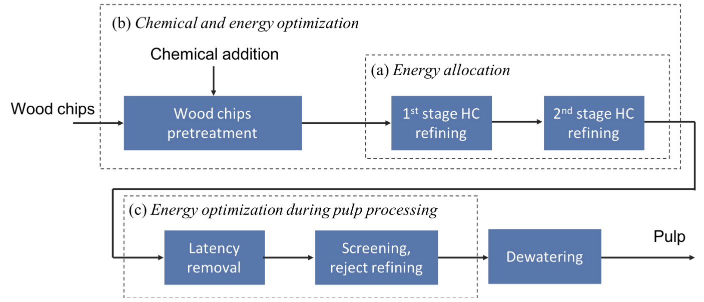

Figure 2. Basic schematic of a two-stage CTMP process (modified from [3]).

(b) Optimization of energy consumption and chemical addition

The addition of chemicals during the wood chips pre-treatment softens the lignin in the wood structure and reduce the energy consumption in the subsequent refining process. We will investigate the connection between chemical addition and energy consumption with the goal of optimizing the process overall operating cost.

(c) Energy optimization during pulp processing

In addition to the two-stage HC refining process, another nonnegligible source of energy consumption is reject refining. The pulp from the reject refining is blended with the pulp from two-stage HC refining for final pulp production during which screening and latency removal procedures are conducted. Existing literature [1,6] indicates that both latency removal and screening processes impact the pulp quality and the energy consumed. We will also optimize the energy consumption during pulp processing.

(d) Hierarchical optimization of the above subproblems

In (a)-(c), individual subunits (or subproblems) are optimized. A two-level optimization framework will be developed to generate an optimal set of operating conditions for the entire pulping process using the optimal solutions for the individual subunits. This will be achieved by utilizing existing optimization tools such as those in [7,8].

(3) CTMP process monitoring

We will develop algorithms for online monitoring of CTMP processes to detect changes in operating conditions, process models, and faults in individual units. These algorithms will detect differences between historical and current operating conditions to better inform operators/engineers [6].

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PROJECT 1.2 (B)

A MACHINE LEARNING METHOD FOR CENTRIFUGAL PUMP PERFORMANCE-MONITORING

Authors: Bryan Bohn, Boris Stoeber, Bhushan Gopaluni

Project Overview

Centrifugal pumps are a fundamental part of fluid transport around the world. Consequently, they are also one of the world's dominant energy consumers. The impacts of inefficient operation, and undiagnosed wear are widely documented and can be disastrous financially, logistically, and environmentally. In this project, we aim develop an affordable, adaptable, and universally accessible sensing method for classifying fluid conditions detrimental to centrifugal pump operation.

Our technique utilizes dynamic pressure measurements, collected at the pump discharge using a single, ordinary pressure transducer. Decomposing these pressure fluctuations into a novel array of statistical features yields characteristic trends correlated to the conditions in the working fluid. These features are then used to train a series of machine learning algorithms, which are in turn used to characterize the adverse conditions.

The diagnostic system presented in this research is unique in that it is not conceived as a standalone tool for pump users, but a shared process between the pump manufacturer and end user. The classification models would be trained and configured by the manufacturer, then provided as a diagnostic service for the operator. The operator need only make dynamic pressure measurements following the manufacturer's method and observe the classification results. In its envisioned application, the scope of the classified phenomena would be augmented by the manufacturer to capture a broad variety of pump behaviors.

Operating Concept

During ordinary operation, the pressure at the discharge of a centrifugal pump is time-varying and cyclic. The magnitude and character of these fluctuations are a function of the pump's configuration, the angular velocity of the impeller ω, the hydraulic load from the surrounding fluid system, and the conditions of the fluid flow. An experimental example for a healthy centrifugal pump with a two-blade impeller is shown in Figure 1.

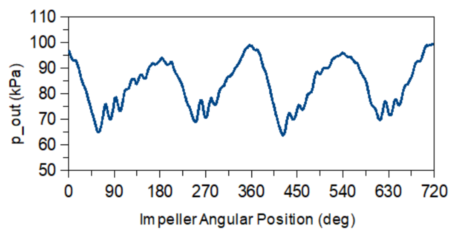

Figure 1. Fluctuations in discharge pressure p_{out} as a function of the angular position of the impeller.

The dominant mode of the oscillation corresponds the impeller's blade-passing frequency (BPF) [1]-[3]. In practice, these fluctuations are often discarded through time-averaging to attain a steady-state measurement. However, the pressure excursions contain relevant information about the pump's fluid behavior. It follows that fluid pressure measurements can be employed to characterize adverse operating conditions.

We test this hypothesis by using dynamic pressure measurements to determine the presence and severity of two detrimental conditions:

a) Gas entrainment – The presence of distributed gas bubbles in the working fluid. Severity is characterized by the *gas void fraction*.

b) Radial impeller wear – Mechanical erosion from the tips of the impeller blades. Severity is characterized by the *impeller loss ratio*.

Prior studies have discussed the dynamic pressure impacts of gas entrainment [4], [5] and impeller wear [6], [7]. Machine learning methods have been proposed as viable means to classify the associated pressure phenomena [8]. In this work, we develop a novel set of statistical measures, called features, to illuminate relevant information about the target conditions. These features then serve as training indicators on which we can create diagnostic machine learning algorithms.

Methods

The studies in this work are conducted in two stages. First, we employ a 2D numerical model (Figure 2a) to generate a small, preliminary set of contrasting severity states for each of the target phenomena. These conditions provides a basis from which to derive relevant measures to quantify the target conditions and propose classification algorithms. In the second stage, we generate comprehensive sets of dynamic pressure measurements for each target condition. Air entrainment data is generated experimentally (Figure 2b). Impeller wear pressure data is simulated using the 2D numerical model. These complete sets of dynamic pressure measurements are then used to demonstrate and validate the classification methods.

PROJECT 1.2 (B)

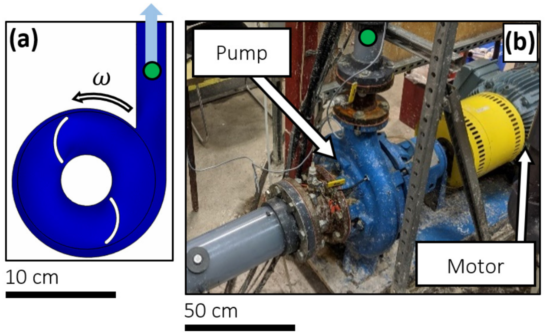

Figure 2. a) A schematic of the 2D numerical pump model; b) The 30 kW pump used as the testbed for the experimental data. In both figures, the pressure transducer position is indicated by a green dot.

Feature and Algorithm Development

We first propose a set of potentially significant statistical indicators. Using the numerical model, dynamic pressure measurements are simulated for healthy, intermediate, and severely degraded states for each target condition. This produces a series of relative trends, which can be used to preliminarily authenticate any meaningful or insignificant correlations between the proposed features and target conditions. An example feature trend from the gas entrainment simulations is shown in Figure 3.

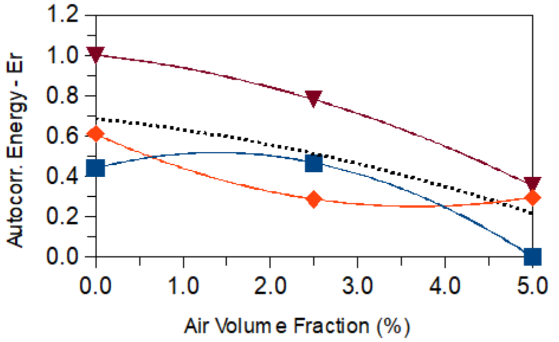

Figure 3. Autocorrelation energy as a function of increasing gas content in the working fluid. The blue, orange, and red indicators delineate slow, intermediate, and high speed states, respectively. The black dotted line is the average. This suggests that as gas entrainment worsens, the relative periodicity of the pressure fluctuations drops. Interestingly, the opposite trend is observed for radial impeller wear.

This initial numerical investigation yields a set of seven relevant features; mean, variance, total signal energy, skewness, kurtosis, total energy of the autocorrelation function, and total energy of the FFT spectrum.

We characterize the states using binary (i.e. healthy or degraded), multi-class (i.e. multiple severity classes ranging from healthy to severe), and continuous-value classifiers. For binary and multi-class classification, a multi-layer perceptron (MLP) with a single, four-node hidden layer is employed. For regression, a Random Forest (RF) algorithm is used. The architecture for the binary classifier is shown in Figure 4.

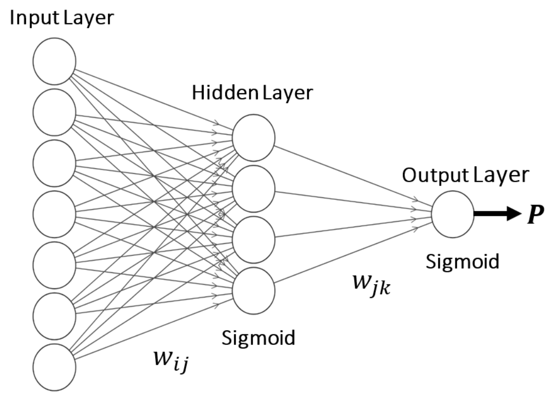

Figure 4. The MLP neural network used for binary classification [9]. The respective weights are indicated by w_{ij} and w_{jk}. The prediction P is either a 0 (healthy) or 1 (degraded condition), based on a severity threshold.

Primary Studies

Dynamic pressure measurements are collected for 310 (experimental) states in the gas entrainment study and 65 (simulated) states in the impeller wear study. In both, half the measurements are employed for training the classification algorithms and half are reserved for testing the classification performance. In both studies, the resulting feature trends follow those of the initial simulations. An example from the gas entrainment study is shown in Figure 5.

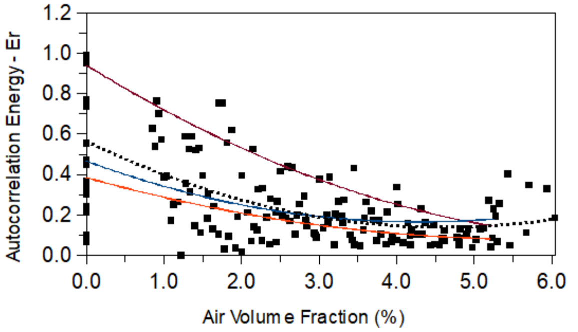

Figure 5. Autocorrelation energy as a function of increasing gas content in the working fluid. The trend agrees with the simulated results in Figure 3. The relative spacing of the trend lines indicates a secondary (undesirable) correlation between the feature and impeller's angular velocity.

PROJECT 1.2 (B)

Classification Performance

In the gas entrainment study, using the base set of seven features, the binary MLP successfully predicts void fractions exceeding 2% in the testing samples with 84% accuracy. The multi-class MLP places the testing states into the correct 1% severity band with 59% accuracy, with 70% of the misclassifications falling just one severity band up or down. The RF regression model has a median error of 0.47 of the true gas entrainment severity percentage.

To improve the prediction performance, an optimization method is proposed that refines the correlations between the features and target condition by reducing their dual dependence on impeller speed. In doing so, the testing performance of the binary and multi-class models is improved to 90% and 62%, respectively. The median error of the regression model reduces to within 0.44 of the true void fraction.

In the impeller wear study, the binary MLP successfully identifies impeller loss ratios exceeding 1.5% in 87% of the testing states, which improves to 97% after optimizing the input features. The multi-class MLP places the testing samples into the correct 1% severity band with 70% accuracy, improving to 82% after optimizing. The median prediction error of the regression model before and after refinement is 0.30 and 0.16 of the true impeller loss ratio, respectively.

Conclusions and Future Work

This research demonstrates that dynamic measurement of the discharge pressure fluctuations from a centrifugal pump is a viable method to evaluate complex fluid phenomena. Using a set of characteristic statistical measures derived from the data of a single low-cost pressure transducer, in conjunction with machine learning, we successfully categorized gas entrainment and radial impeller wear. There are no present commercial devices that offer comparable cost or resolution.

There are a variety of avenues by which the research could be augmented. First, it is recommended that trials be conducted to validate the impeller wear classification method using experimental measurements. Secondly, though our research focuses specifically on gas entrainment and impeller wear, there is a potential to expand the approach to include a variety of other fluid phenomena, adverse or otherwise. It would be valuable to investigate the full spectrum of fluid behaviors that can be characterized using this technique, and explore the statistical means necessary to observe and characterize their correlations.

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PROJECT 1.3

CREATING LOW ENERGY SHIVE-FREE PULPS

Authors: Rodger Beatson, Heather Trajano, Sudipta Kumar Mitra, Claire Maulit

Background

The objectives of this project are to: (1) generate a better understanding of the impact of chemical/biological treatments on development of fibre and fines properties during LC refining, and (2) develop economically viable low-energy processes that combine the use of such treatments with LC refining for the production of printing/writing and board grades.

Highly alkaline peroxide treatment (HAPT) of mechanical pulp is well documented in our previous work. This project is an extension of previous work on the use of highly alkaline peroxide treatment in the production of mechanical pulp [1, 2]. That highly alkaline peroxide treatment reduces the energy required to obtain a given tensile strength was established in these studies. Introduction of a two-stage process overcomes the brightness drop due the high alkalinity while still increasing strength of pulp. In this two-stage process all the peroxide is added in the first stage which is operated at lower alkalinity and extra alkali is added in the second stage operated at a lower temperature.

Recently, in an effort to obtain a better understanding of the mechanism behind the strength gains in HAPT, we treated second-stage TMP, using a one-stage HAPT process, and determined the effect of varying the alkali charge on the settling rate, and handsheet structure and properties.

Experimental

A secondary refiner TMP (60% Spruce/40% Pine) of CSF 390 ml was treated with 4% hydrogen peroxide while varying the alkali charging from 2.5% to 6% as shown in figure 1. After washing with deionized water, pulp properties were evaluated. For better understanding of the effect of alkaline peroxide treatment on mechanical pulp, a pulp suspension test at 0.3 % consistency was performed for each sample, to monitor the settling rate and end settling height of treated pulp suspension. Handsheets were made to measure strength and optical properties of paper.

Results

Figure 2 shows the impact of different alkali charge on strength and brightness of TMP with peroxide treatment. We see a consistent rise in strength of paper with increasing alkali charge whereas increase in brightness remains constant. A single stage peroxide treatment with 2.5% alkali charge increased brightness by 40% as expected from previous work. It also increased tensile strength by 40% from untreated pulp which is higher than previous studies. As we increase the alkali charge to 4%, we see a higher increase in tensile strength up to 60% and with no benefit in brightness. Further, 6% alkali charge showed higher tensile gains without the drop in brightness seen in previous work. Tensile strength almost doubles at the alkali charge of 6% confirming that highly alkaline peroxide treatment can be used to produce high strength mechanical pulp.

The rise in strength of mechanical pulp by HAPT process is well known and documented, but the mechanism of strength development is not clear. For better understanding of this mechanism we performed a simple suspension test of our untreated and treated pulp. We took 1000 gm of pulp suspension at 0.3 % consistency, mixed well and poured in a 1000 ml graduated cylinder. Then we let pulp to settle until final settling height reached. From figure 3, it can be seen that end settling height of pulp suspensions decreases significantly with increase in alkali percentage along with an increase in tensile strength. The pulp fibres in suspension are consolidating under the influence of gravity. We hypothesize that this chemical treatment is modifying the fibre surface in such a way that the attractive forces between the fibres are increased. This may be related to the generation of acidic groups on the surfaces of the fibres and fines as previously reported in [2].

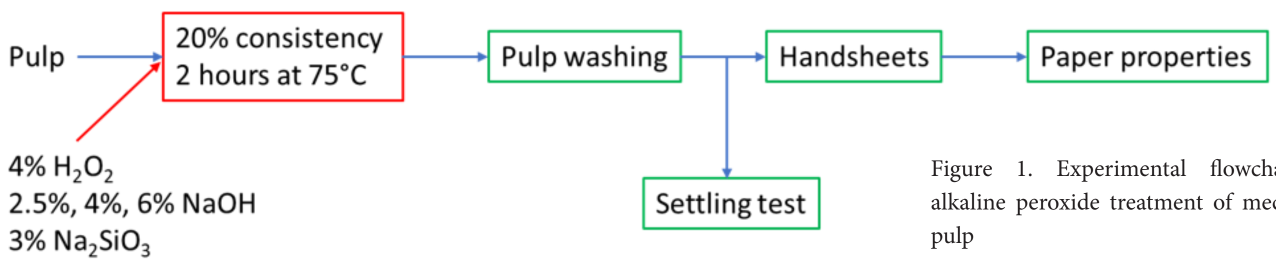


Figure 1. Experimental flowchart for alkaline peroxide treatment of mechanical pulp

PROJECT 1.3

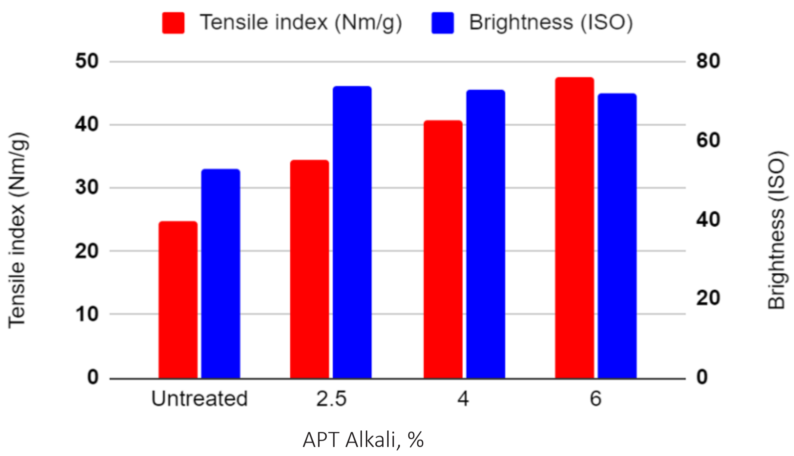


Figure 2. Effect of alkaline peroxide treatment with varying alkali charge on tensile strength and brightness of paper.

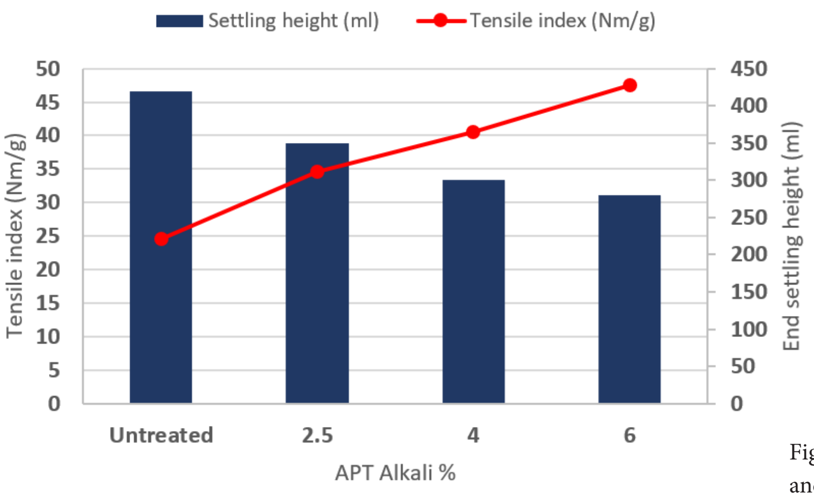
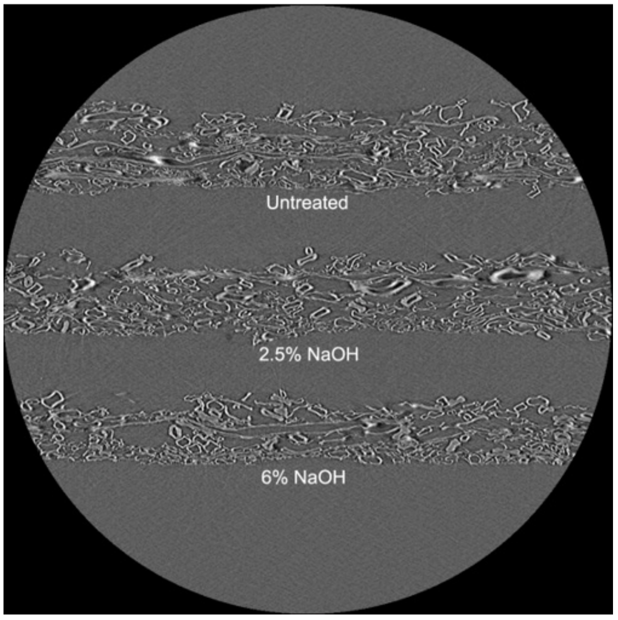


Figure 3. Comparison of tensile strength with changes in settling height with increasing alkali charge in alkaline peroxide treatment. Red line indicates tensile strength increase.

Figure 4. X-ray tomography image of cross section of untreated and Alkaline peroxide treated handsheets (1.25μm resolution)



For better understanding of the phenomenon and the driving force which significantly increases the strength of paper made with TMP treated by the HAPT process, we are studying pulp and handsheets by microscopy. Figure 4 shows a preliminary image, generated by X-ray tomography, of cross sections of handsheets of untreated and treated samples. We see clear differences in bulk and consolidation of pulp fibres/fines of untreated and treated samples. As we move from untreated to lower alkalinity (2.5%) the density of fibre/fines increases, and the increase is even greater as the alkalinity increases. This consolidation of fibres parallels the settling of the pulp suspension shown in figure 3. There is a noticeable increasing density gradient from top to bottom within the handsheets made from the treated pulps, this must be related to consolidation of fibres/fines due to high alkalinity, but the exact mechanism is the subject of future research.

PROJECT 1.3

Conclusion

High alkalinity peroxide treatment of mechanical pulp can increase the tensile strength without dropping in brightness. The lack of a brightness drop contradicts some of our earlier results and requires further investigation. This process can be used for producing stronger pulp for grades of paper which demands high strength like flexible packaging paper grades. A simple settling test and imaging by X-ray tomography provide insight into the effect of the highly-alkaline peroxide treatment on fibre and sheet consolidation. Pulp fibres and fines consolidation clearly plays an important role in the mechanism of strength development, though further studies needed.

Future Research

In future work we will investigate the role of the HAPT fines in sheet consolidation and the effect of the HAPT treatments on the response of the handsheets to the pressing process.

Other work under this project will evaluate the various chemical treatments explored in Phase I and Phase II of the ERMP program with respect to their suitability for use in different products such as paperboard, printing papers and flexible packaging.

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PROJECT 2.1

LIGNIN RICH FINES: SIMPLE ROUTES TOWARDS CREATION OF HYDROPHOBIC AND HYDROPHILIC FILLER ADDITIVES

Authors: Scott Renneckar, Liyang Liu, Siwei Chen

Background

Thermomechanical pulp fines (particle size < 75 um) comprised as high as 30% of the weight of TMP in some cases [1]. These particle materials are significantly smaller than the bulk fibers and have a different composition, e.g., a higher concentration of lignin (35% - 40%) and more extractives on their surface. Due to the heterogeneous character and small size, these fines will impact the mechanical properties of final paper sheets as their concentration increase. Consequently, the thermomechanical pulping and paper making process may lose these small particle materials in the waste stream rather than capture them as valuable co-products. Taking the benefit of their size, our project aims are directed towards using a greener surface modification route to tune the properties of these fines, including the surface energy (hydrophobicity vs. hydrophilicity), thermal properties, and reactivity. In so doing, these small fines may be of interest to make more valuable polymer blends as 3D printing materials or enhanced hydrophilic additives for handsheets.

Recently, additive manufacturing combining computer-assisted design (CAD) is a promising versatile platform to rapidly manufacture 3D objects, even at home. This technique can be more flexible and economical than conventional processes in creating innovative structures (e.g., subtractive, molding, and casting). In last 10 years, the overall market in 3D printing materials is growing rapidly [2]. Consumers are looking for natural products such as wood-based materials. Further, fines may be an ideal additive for this process. Their small particle size will help prevent nozzle clogging that can typically occur when processing conventional wood filaments (containing 30% of wood fibers and 70% polymers). Moreover, the surface energy and thermal properties will play prominent roles in converting fines to plastic additive. Surface chemical modification, especially esterification, is critical to modify the hydroxyl rich fibre surface and tune their thermal properties. Previous researchers have reported several interesting esterification approaches on lignocellulosic materials. Suzuki et al. recently adopt a one-pot, two-step homogeneous transesterification method to esterify the surface hydroxyl groups with mixing decanoyl and acetate groups, [3]. Their process successfully converted sugarcane bagasse to injectable thermoplastics and realized the separation of lignin from parent materials simultaneously. Gregorova et al. adopted stearic acid to treat wood products, then were blended with degradable aliphatic polyesters [4]. The resulting materials showed promising thermoplastic properties and comparable mechanical properties to similar materials without natural fibre.

On the other hand, the functionalization of fines with hydrophilic groups, such as amines or carboxylic acids, may be of interest in conversion to enhanced additives for handsheets. Seelinger et al. utilized hydroxypropyl cellulose (HPC) as an enhanced additive through a novel crosslinking process. TEMPO-mediated oxidization (2,2,6,6-tetramethylpiperidinyl-1-oxyl) was applied to modify the secondary aliphatic hydroxyl to ketone groups. Following the addition of amines-based crosslinkers, this paper sheet with oxidized HPC showed improved wet resistances [5]. As a result, with more reactive functional groups and charges, these surface modified fines may serve as enhanced additives in the hand sheet and they will better interact with mineral additives or cationic polyelectrolytes.

We currently have two objectives:

a) Adopting the direct esterification to tune the thermal properties of fines with different types of an organic acid, [6]

b) Choosing suitable routes to obtain hydrophilic fines with either amine or carboxylic acid groups

Results

Our initial results adopted the bleached fines as starting materials, containing over 95% water and a negligible amount of lignin. The lyophilization or solvent exchanges were required as the first step to prepare mixtures suitable for the subsequent modification. To prepare hydrophobic fines, the direct esterification using propionic acid as solvent, reagent, and catalyst was applied to modify the hydroxyl groups in the dried fines. FT-IR results (Figure 1) showed the modified products contain ester peaks (C=O, 1732 cm-1) and have a slight reduction of hydroxyl group peak (O-H, 3340 cm-1) indicating it was heterogeneous conversion without dissolution of the fines into the mixture.

PROJECT 2.1

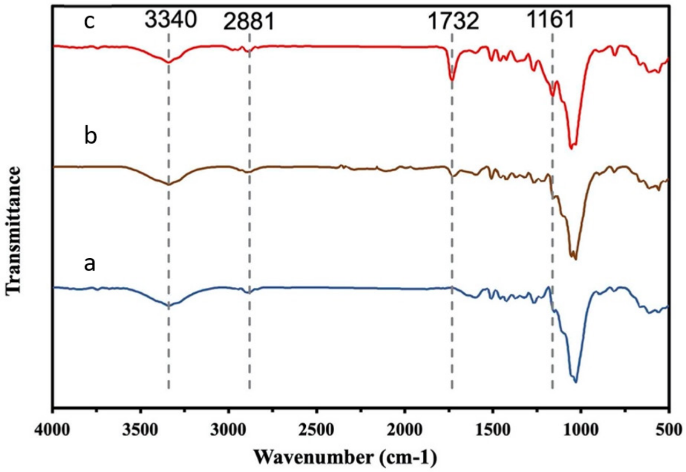

Figure 1. FTIR spectra of bleached fines before (a), after glutaric anhydride esterification (b), and after the direct esterification with propionic acid (c).

To make hydrophilic fines, glutaric anhydride esterification route was aimed at adding carboxylic acid groups. This route unfortunately required pyridine as catalyst and co-solvent and the method was used as a benchmark. Although the FTIR of modified products (Figure 1b) showed carbonyl peaks, their small amounts indicated low efficiency of this route. Another promising route in preparing hydrophilic fines is TEMPO-mediated oxidation. With this technique, solvent exchange or lyophilization are unnecessary; this modification route can be performed in alkaline water (pH=10). To monitor modification, conductometric titration was used to quantify total carboxylic acid groups. Our initial results showed high efficiency of this modification method, as the appearance of plateau area in their conductivity titration curve associated with carboxylic acid groups (Figure 2). This plateau can be readily converted to the mmol of acid per gram of fibre.

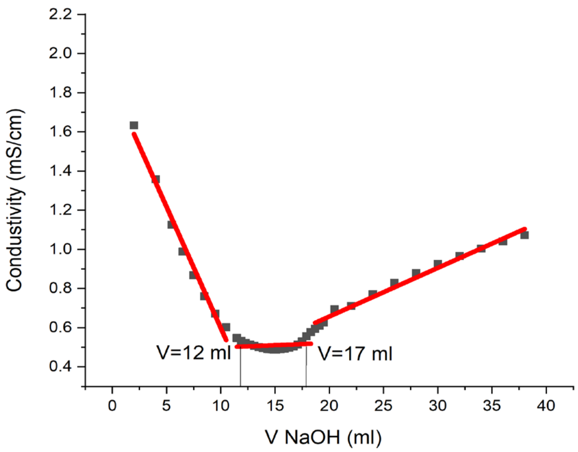

Figure 2. The conductive titration curves of TEMPO oxidized leached fines.

Future Research

We recently received the primary fines containing a higher amount of lignin and extractives. All our studies will start from these products. Our next stage work will include but not limit to:

a) Understanding the fundamental composition (lignin contents and moisture) and thermal properties of these fine products;

b) Investigating potential solvent to disperse these fines or exchange it with the organic acid for the subsequent esterification

c) Optimizing the reaction conditions of the esterification route to prepare fines with different thermal properties

d) Blending the modified fines with typical thermoplastic materials (e.g., PLA or PHB) and evaluate the compatibility of various fines in light of their mechanical properties

e) Starting with bleached fines, optimize the glutaric anhydride route and tempo oxidization route

f) Investigating the potential a new route to add more hydrophilic amine groups on the surface of fines

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PROJECT 2.2

FROM TREES TO TREATMENT FUNCTIONALIZING TMP EXTRACTIVES

Authors: Pierre Betu Kasangana, Cameron Zheng, Laurel Schafer, Heather Trajano

Background

Wood extractives are non-structural low-molecular-weight compounds. They can be found as free molecules or bound in glycosides but they can also be found in association with the structural polymers (cellulose, hemicellulose, lignin) [1]. They may be linked by low energy intermolecular interactions to a structural polymer or simply deposited in the lumens of wood cells and other structural cavities. For these reasons, they can easily be solubilised by various solvents (e.g. water, ethanol, hexane) [2].

Depending on their polarity, wood extractives are classified as hydrophilic or lipophilic. Lipophilic wood extractives mainly include resin acids, mono/sesquiterpenes, long chain fatty acids and alcohols, sterols, waxes, sterol esters, and triglycerides, which make up less than 3% of the dry wood mass [3]. Due to their nonpolar nature, lipophilic extractives are hard to eliminate during pulping and bleaching. A majority of the compounds are removed during debarking, pulping, bleaching, washing, and with the final product, paper. Unremoved extractives remain in the final product, disturb further processing of pulp and lower the product quality [4].

Since November 2020, the primary objective has been to analyze the extractives in process samples from Meadow Lake Pulp Mill.

Experimental work

Thirteen samples were obtained from Meadow Lake Pulp Mill as summarized in Table 1. The samples were collected on Day 5 of a run processing aspen. Samples of aspen chips (two suppliers: ASP and MLPM), softwood, and softwood bark were provided. The fourteen samples were divided into three groups in this study. Chip and bark samples (ASP, MLPM, SWD, BARK) are in the first group, solid pulp samples (N21, 23, 30) are in the second group, and liquid samples (N9, 17, 19, 22, 31, 32) are in the third group (washing water, bleach, and condensed vapours). The carbohydrate and lignin compositions of chip samples and bark were determined according to Sluiter et al. [5] The total extractive content of chips samples was determined using accelerated solvent extraction (ASE) system with deionized water followed by ethanol. The liquid samples were subjected to liquid-liquid extraction with methyl tert-butyl ether (MTBE) extracts prior to characterization with gas chromatography-mass spectrometry (GC-MS) and high-performance liquid chromatography with photo diode array and mass spectrometry (HPLC-PDA-MS). Fines were recovered from the pulp samples using Dynamic Drainage Jar (DDJ) fractionation as described by Kangas et al [6]. Prior to fractionation, TMP pulp was hot disintegrated (85ºC, 10 min.) and diluted to 0.5% consistency with distilled water. The pulp slurry was passed on to the DDJ apparatus, which was equipped with a 200 mesh (76 μm) wire and propeller stirring, and the valve was opened. The suspension was washed with distilled water until 10 liters of slurry had been collected. The fines fraction passed through the wire while the fibers were retained on the screen.

Table 1. Process Samples from Meadow Lake.

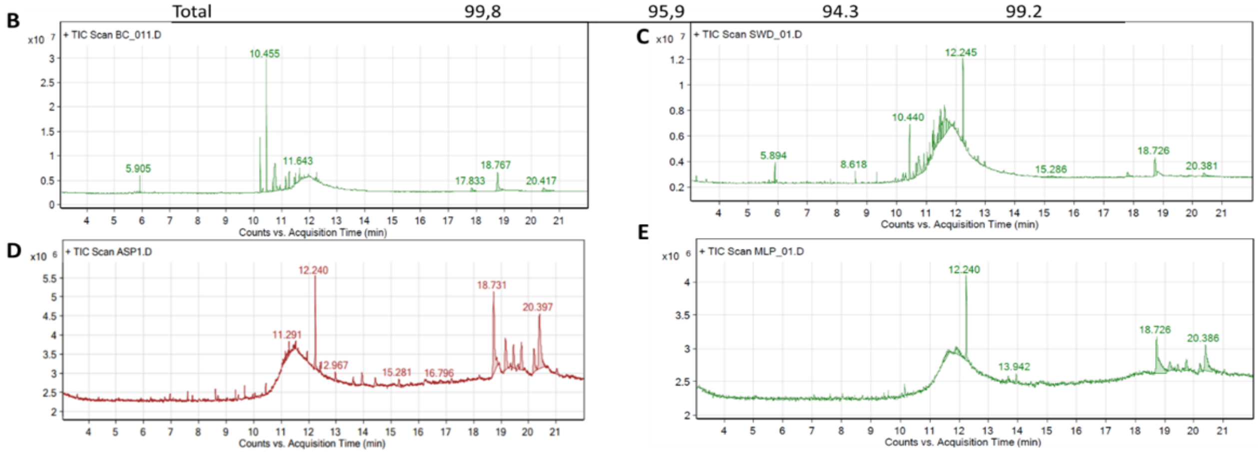
Sample ID	Sample points	pH	Total Solids (%)
9	Front end purge to water recovery sump	6.09	3.04
17	Final cleaner rejects	6.61	3.47
19	L1 Clear white water	6.39	3.31
31	2% Evap Feed	7.50	10.40
32	30% Evap Out	7.05	24.27
21	Interstage Presses (ISP) Pulp Out	Solid	
22	ISP Filtrate	6,5	3.29
23	P2 Twin Roll Press (TRP) Pulp Out		
30	Final TRP pressate	Solid	
Softwood Chips (SWD)	Carrier	Chips	
Aspen CHIPS (MLMP)	Carrier	Chips	
Aspen Chips (ASP)	Carrier	Chips	
Softwood Bark	Carrier	Bark	

PROJECT 2.2

Figure 1. Chemical composition of chips and bark samples (A) as well as their hexane extract profiles in GC-MS(B-E)

A

Main constituants	Softwood Bark	Aspen Chips (ASP)	Aspen Chips (MLMP)	Softwood Chips (SWD)
Glucose	31,4±3,2	49,6±3,3	48,8±1,3	53,5±3,2
Xylose	9,5±1,9	17,7±0,1	18,3±1,3	14,7±1,5
Arabinose	10±1,1	ND	ND	9,2±1,3
Lignin total	26,8±2,3	23,9±0,7	22,9±2,5	18,6±3,2
Insoluble lignin	24±1,1	20,5±0,2	21,5±1,2	16,8±1,9
Soluble lignin	2,8±1,2	2,4±0,5	2,4±1,3	1,8±1,4
Ash	3,1±1,4	0,8±0,01	0,8±0,05	0,3±0,06
Total extractives	19±2	3,9±0,3	3,5±0,7	2,9±0,7
Total	99,8	95,9	94.3	99.2



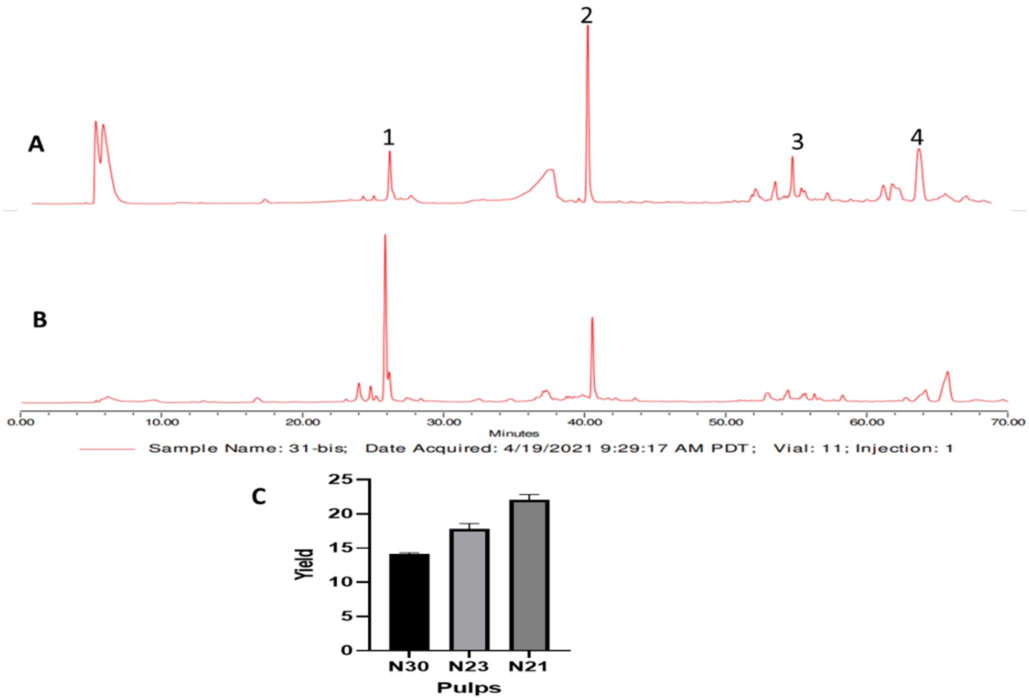
Preliminary results

As shown in Fig 1A, the softwood (SWD) sample is in lignin and extractives compared to aspen (ASP and MLPM). Softwood bark has the highest concentration of lignin and extractives, as expected. Due to its high extractive content, hexane extract of bark was used to optimize the method of identifying extractives by GC-MS and HPLC-PDA-MS. The hexane extract of each chips and bark was prepared using the ratio of 1.5g dry sample/ 15 ml solvent, by continuous shaking (250 rpm) in an orbital shaker at room temperature for 24 h. Bark had the highest extractive yield(3.06 %), followed by softwood (1 %), MLPM (0.85 %), and ASP (0.66 %.) As shown in the GC-MS chromatogram in Table 1B-C, the bark and softwood hexane extracts have approximately 3% and 0.7% monoterpenes, respectively. The identified monoterpenes are α-pinene (peak at 5.9 min) and α-terpineol (peak at 8.6 min). In contrast, hexane extracts of MLPM and ASP primarily contain diterpenes, which will be identified and confirmed in further studies. Interestingly, the hydro-distillation of SWD samples yielded 0.016% essential oil, which is around two times lower than the average yield of essential oils in softwood bark (0.04-1%) [7].

Fig. 2 A-B presents the HPLC-PDA profiles of MTBE extracts obtained from clear white water (N9) and the 2% Evap. Feed (N31). In all MTBE extracts of liquid process samples, peaks were consistently identified at 26.1, 40.5, 55.4 and 64.6 minutes. With the exception of the peak at 26.1 minutes, all peaks likely correspond to lipophilic compounds due to lipophilic nature of the mobile phase at those retention times. The N19 and N31 extracts were less complex than the other liquid samples (not shown) therefore these two extracts will be investigated further to identify and isolate the main components. The first step for pulp analysis was to use the DDJ method to separate the fibres and fines. As shown in Fig2.C, the yield of fines in samples N21, 23, and 30 is 10%, 14% and 24%, respectively.

PROJECT 2.2

Figure 2. HPLC profiles of N19 (A) and N31 (B) samples as well as the yield of fine materials (C) in N21, 23 and 30 pulps.



Future Research

The next milestone will be to finalize the extractive profiles of liquid samples by identifying and characterizing the primary components by using chromatographic, spectroscopic and spectrometric methods. The identification and quantification of the extraction of different samples will enable us to determine the most promising liquid samples for future valorization. The lipophilic fraction will be delivered to Professor Schafer's laboratory for hydroaminoalkylation (Fig.3).

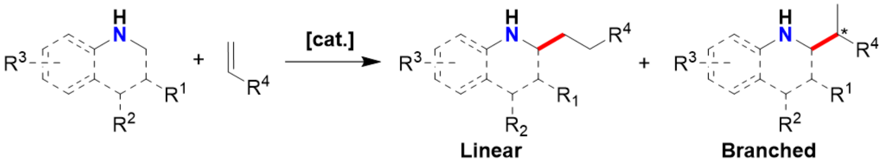


Figure 3. General reaction scheme for hydroaminoalkylation.

Compositional analysis of the pulps will continue. This analysis will support efforts to resolve current process concerns at Meadow Lake and future valorization efforts. Central-composite design of experiments will be used to determine the best conditions for extractive desorption from fines recovered from the pulps. Solid consistency, slurry temperature, electrolyte addition (type and concentration), pH and residence time will be factors in experimental design. To ensure mass balance closure, solid and liquid samples will be examined before and after desorption.

The next step will be to characterise the extractives adsorbed on fines. The HPLC and GC-MS profiles of extracts of fines will be compared to that of liquid samples to identify major compounds; we expect to find resin acids and terpenes. Principal Component Analysis (PCA) will be used to illustrate the variability of extractive composition in the samples as a result of changing parameters during the chemometric analysis.

PROJECT 2.2

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PROJECT 2.3

MFC PRODUCTION, CHARACTERIZATION, AND PROPERTIES OF MECHANICAL PULPS

Authors: Jingqian Chen, Mariana Frias de Albuquerque, Samira Gharehkhani, Reanna Seifert, James Olson, Boris Stoeber, Heather Trajano

Background

This project aims to produce Micro-Fibrillated Cellulose (MFC) from mechanically fractionated fines. In previous work it was demonstrated that LC refining of mechanical pulp can produce MFC capable of reinforcing TMP sheets. In this project we hypothesize that the natural microstructure of the 'fines' subject to mechanical and enzymatic treatment will disassemble into an MFC suitable for reinforcement of high bulk, long-fibre TMP sheets, and that this MFC can be applied to the conventional TMP sheets to improve strength, surface, and barrier properties. Staffing of this project has been completed. The final team member, Dr. Rasmita Sahoo, will arrive in Canada in June 2021.

Enzymes for MFC Production

Many types of enzymes have been applied for MFC production: endoglucanases, cellobiohydrolases (exoglucanases), β-glucosidases (cellobiases), and hemicellulases (Nechyporchuk and Belgacem 2016). Treatment with endoglucanase causes fiber length and degree of polymerization to decrease (Tian et al. 2017; Henriksson et al. 2007; Long et al. 2017; Siqueira et al. 2010) and cellulose crystallinity to increase (Long et al. 2017; Nechyporchuk et al. 2015). Endoglucanase pretreatment produced MFC nanofibers with high aspect ratio (length to diameter), a key parameter for reinforcement applications (Pääkkö et al. 2007; Henriksson et al. 2007). Endoglucanase has been used in combination with other cellulases. Siqueira et al. (2010) found that the application of endoglucanase produced a mixture of MFC and rod-like cellulose fibers while the use of exoglucanase preserved the MFC structure but failed to produce high-aspect ratio fibres.

Another family of enzymes to support MFC production are hemicellulases such as xylanase and mannase. Removal of hemicellulose may reduce the fibre integrity, increase the accessibility of cellulose fiber to cellulases and facilitate subsequent mechanical fibrillation (Long et al. 2017). Tian et al. (2017) examined the effects of a cocktail of mannase and xylanase and subsequent mechanical refining on Northern Bleached Softwood Kraft pulp. Microscopic imaging of the hemicellulase-treated material revealed that the helical twine-like structure of fibrils gradually uncoiled as the number of refining revolutions increased resulting in high specific surface area, a key attribute for the use of MFC in reinforcement applications. Hu et al. (2011) observed that simultaneous application of xylanase and cellulases increased average fiber width by approximately 30%. As fibre width is indicative of fiber swelling, simultaneous use of cellulase and xylanase may improve fibrillation. Long et al. (2017) tested cellulases in combination with four different commercial xylanase products. The addition of xylanase to cellulases increased the degree of fibrillation but the xylanase products did not perform identically.

Varying enzyme type, dose, and treatment conditions will produce MFC with varying properties. The literature does not provide clear guidance on how to manage these variables in order to target production of MFC with specific properties. Thus, it is necessary to conduct a parametric study to better understand enzymatic hydrolysis in order to produce homogeneous, high quality MFC.

Assessing Changes in Fibre Characteristics with Gel Point

Quantifying pertinent MFC properties such as bulk, tensile and tear index is important for comparison purposes. However, these properties are generally obtained from handsheets (Ehman et al., 2020; Jahangir & Olson, 2020). Handsheet preparation is time and labour intensive, therefore a rapid method for assessing fibre properties is needed to support the planned enzyme screening studies. One possible approach is measurement of gel point concentration. Martinez et al. (2001) defined gel point concentration (Φ_g) in a fibre suspension sedimentation as the lowest concentration at which the network can support load, i.e., the lowest concentration at which the fibres form a continuous network. Cellulose fibre aspect ratio is known to influence hand sheet properties therefore Varanasi et al. (2013) developed methods to estimate aspect ratio from gel point. The sedimentation test consists of preparing 250 mL of fibre suspensions with different initial concentrations in graduated cylinders and observing the ratio between suspension height and initial height (H/H0) for 48 hours. A graph of initial concentration versus H/H0 was plotted and fitted with a second order polynomial; the gel point is the linear coefficient of this function. It is hypothesized that measuring gel point or aspect ratio can be used to rapidly evaluate fibres subjected to enzymatic pre-treatment.

PROJECT 2.3

Results

MFC Production through Screening and Refining

The initial stage investigated the impact of mechanical refining without enzymatic treatments. Bleached chemi-thermomechanical pulp was fractionated via pressure screen in 2 trials (0.5mm and 0.8mm screen holes). The resulting short fibre streams were dewatered to 3.5% consistency and refined into MFC. The refined short fibres were re-mixed with their respective unrefined long fibre counterparts at different ratios (0 to 100% MFC content) to compare with whole pulp refined without fractionation.

Fractionation alone created short fibres with a bulk-freeness matching the highest refined points of the whole pulp regardless of the screening basket used (Figure 1). The 0.5mm holed screen produced short fibres with a tensile strength comparable to whole pulp refined at ~250kWh/t. Once the short fibres were refined, the bulk-tensile curve of the 0.5mm screen out performed the 0.8mm trial but fell short of the refined whole pulp. The unrefined long fibres, regardless of the screening basket used, had elevated bulk and freeness values compared to the whole pulp.

The unrefined long fibres were incorperated with the refined short fibres at different mass ratios (Figure 2). For most points the bulk was elevated compared to the refined whole pulp at any given freeness value for both screening trials. In terms of bulk-tensile, composites at the mass reject ratio closely matched refined whole pulp. Higher precentages of long fibres yielded higher bulks, most notably in the 0.8mm trial. Higher percentages of short fibre MFC reduced both bulk and tensile index compared to refined whole pulp. These results illustrate how fractionation alone, whole pulp MFC and short fibre MFC can provide unique benefits. The data will provide a baseline to quantify the effects of adding enzymatic treatments and identify the circumstances in which enzymes have the greatest impact.

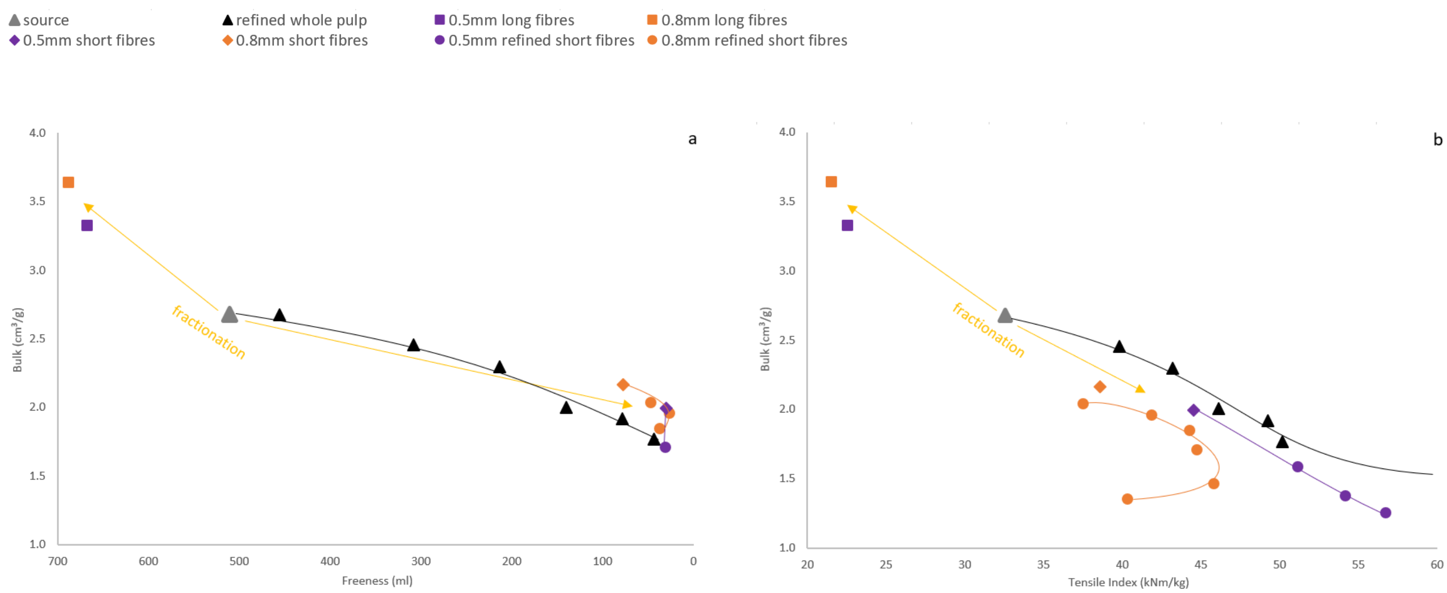

Figure 1. Fractionation and short fibre refining only. Black line represents refined whole pulp. a) Bulk (cm^3/g) vs Canadian Standard Freeness (ml) b) Bulk (cm^3/g) vs Tensile index (kNm/kg).

PROJECT 2.3

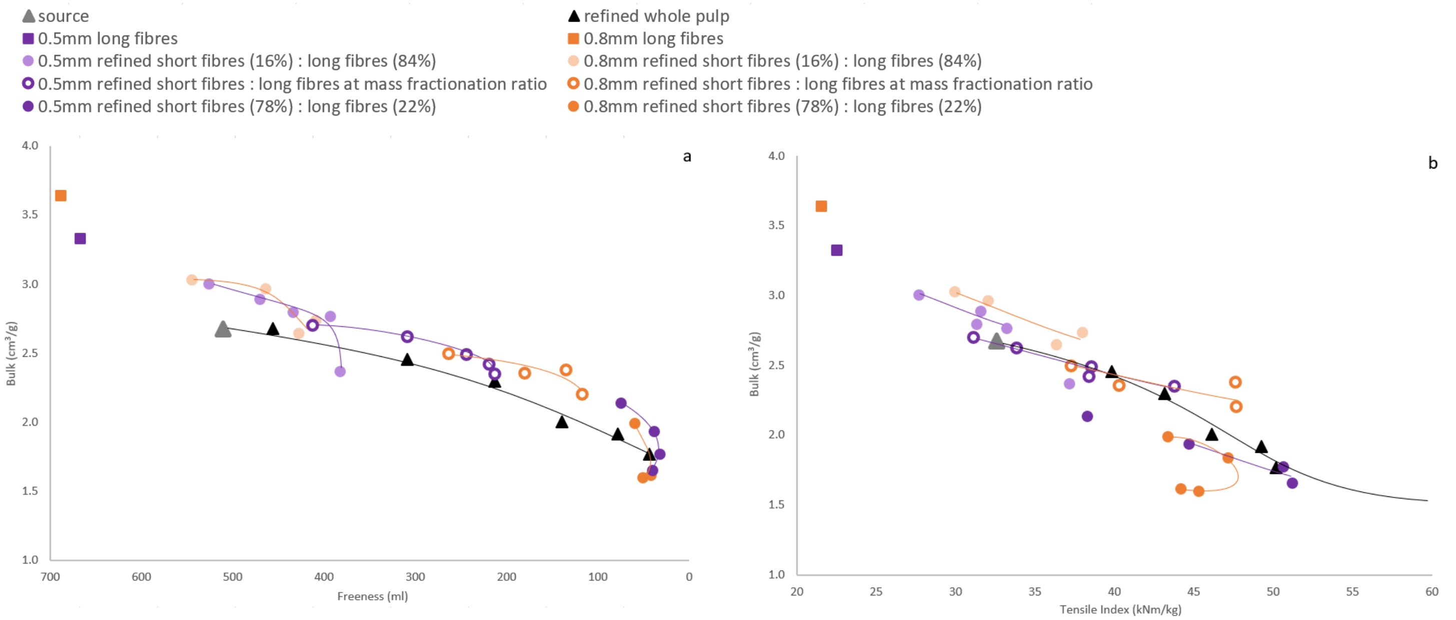

Figure 2. 0.5mm and 0.8mm short fibre MFC mixing with long fibres at 16%, mass reject ratio of the initial screening and 78% refined short. Black line represents refined whole pulp. a) Bulk (cm^3/g) vs Canadian Standard Freeness (ml) b) Bulk (cm^3/g) vs Tensile index (kNm/kg).

Assessing Changes in Fibre Characteristics by Gel Point

The gel point concentration point (Φg) was determined for short fibres (obtained using a 1.0 mm holed screen) subjected to different refining levels. The gel point concentration increased with refining between 0 and 420 kWh/t (Table 1). Properties of handsheets prepared using the short fibres were measured. Bulk, tear index, freeness and average fiber length decreased with increasing gel point, while tensile index and fines content increased with increasing gel point (Figure 3).

The sedimentation test was also performed for short fibres subjected to refining at 700 kWh/t. These fibres displayed unique behavior. Sedimentation occurred more slowly; it was not possible to definitively determine gel point after 48 hours. Sanchez-Salvador et al. (2020) reported similar observations and attributed it to the high charge of highly refined fibres. The authors proposed adding salts to the suspension or adjusting the pH to reduce the fibre charge and accelerate settling.

Table 1. Gel point concentration (g/L) for fibre suspensions of different refining levels of short fibres fractionated from a 1.0mm holed screen.

SRE (kWh/t)	Gel point (g/L)
0	4.2
217	6.8
420	7.9

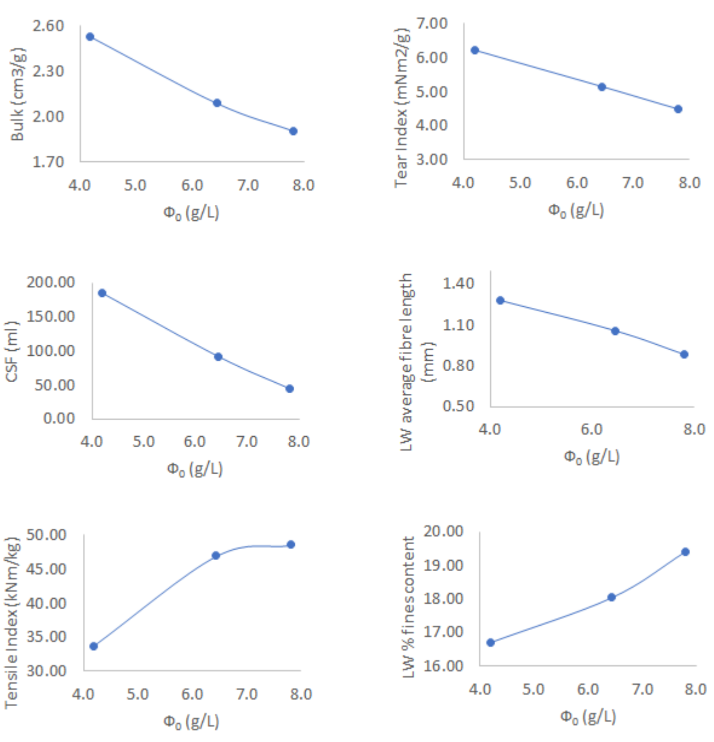

Figure 3. Variation of bulk, tear index, freeness (CSF), average fibre length, tensile index and fines content of refined short fibres with measured gel point.

PROJECT 2.3

Future research

Enzymes for MFC Production

Enzymatic hydrolysis of four types of enzymes (endo-glucanase, exo-glucanase, xylanase and mannanase) will be assessed to determine the performance on TMP to produce a low-grade MFC. Sugar and lignin composition will be determined by NREL LAPs. Free enzyme concentration will be determined by the intrinsic protein fluorescence by UV-Vis spectroscopy. The PFI mill will be used to refine the TMP fibre with narrow gap size according to TAPPI standard T248 sp-00.

Assessing Changes in Fibre Characteristics

1. Gel Point

The determination of the gel point through sedimentation tests is suitable for lightly refined fibres (below 700 kWh / t). At higher levels of refining, alternate techniques are required. Possible alternatives include adding salts, adjusting pH and use of a Turbiscan. Another possibility is to calculate gel point through the analysis of yield stress.

Yield stress is the stress at which a medium adopts continuous strain at constant stress (Kerekes, 2006). The yield stress, determined by the vane method, can be used to identify the transition between dilute fibre suspension to semi-dilute fibre suspension and can be interpreted as the suspension gel concentration point. We believe that the same technique can be used to establish the relationship between morphology and rheology of MFC, providing an opportunity to monitor MFC production. In the next step, a relationship between the aspect ratio and gel concentration point will be established using the crowding number theory developed by Kerekes (Kerekes & Schell, 1992).

2. Visualization

Fluorescence labelling technique have been used for wood particles and pulp fibres for morphology and enzyme interaction analysis by confocal laser scanning microscopy (Helbert et al. 2003; Luterbacher et al. 2015; Long et al. 2019; Imai et al. 2019). Combining this technique with cross- section and 3D image analysis could enable MFC fibrillation and fines structure analysis and offer insights on morphology variation with enzymatic and mechanical treatments.

The Nikon Eclipse Ti-E microscope will be used to analyze the fibrillation and the fibre morphology of TMP MFC in the future trials. The fibre cellulose will be labelled by 5-([4,6-Dichlorotriazin-2-amino) fluorescein hydrochloride (DTAF).

Visualization with TEM, SEM and microCT will also be explored.

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PROJECT 2.3

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PROJECT 3.2

ADVANCED CHARACTERIZATION – COMPUTED TOMOGRAPHY

Authors: Aurélien Sibellas, James Drummond, Mark Martinez, André Phillion, Mengqi Feng, Jingqian Chen, Rodger Beatson

Background section

X-ray microcomputed tomography is a powerful non-destructive technique to acquire 3D images of objects at the microscopic scale [1]. As the microstructure of the wood and paper handsheets studied in the ERMP consortium plays an important role in controlling properties, 3D imaging with tomography is a natural choice to probe their inner structural arrangement. Over the past six months, we have used X-ray tomography to examine:

a) The effect of LC refining and/or MFC addition on the microstructure of mechanical pulp handsheets.

b) The effect of chemical impregnation and/or axial compression on the microstructure of wood chips.

Project 1 – Imaging of Mechanical Pulp Handsheets

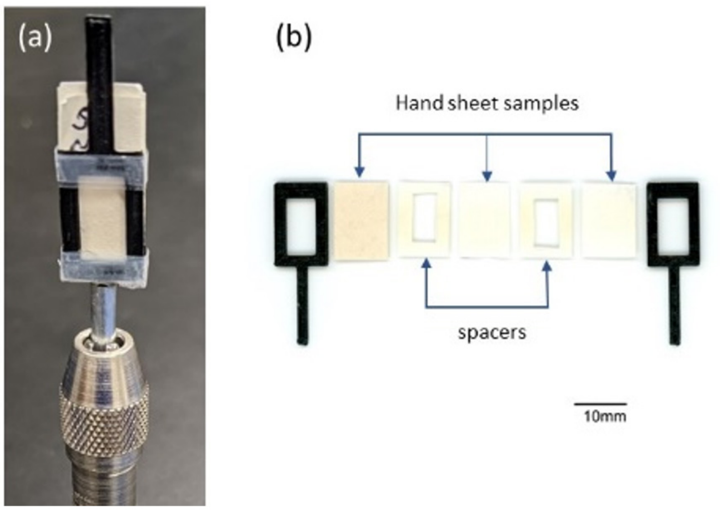

Figure 1. (a) Paper handsheets in the sample holder (black) mounted on the pin vise mount of the X-ray tomograph. (b) Example of the arrangement of multiple samples within the sample holder. Tape is used to hold the assembly together.

The vision of this project is to gain insight into the microstructure of mechanical pulp handsheets at different MFC and LC refining levels. The first step in this process was to develop an effective sample holder. We designed a sample holder for imaging multiple sheets simultaneously to increase throughput (Figure 1). It comprises 3D-printed frames housing multiple paper samples separated by gaskets/spacers. We are able to house up to 5 samples in the holder.

The second step was the development of an imaging protocol that balances resolution, sampling time, and image quality. Using a High Aspect Ratio Tomography (HART) imaging protocol, we performed a systematic study to optimize scanning parameters to resolve features of the fibre wall. With this protocol we examined the structure of CTMP handsheets with various levels of MFC addition. One representative image of a 60 g/m2 CTMP handsheet is shown in Figure 2a. Representative cross-sectional images of handsheets with different levels of MFC content are shown in Figure 2b.

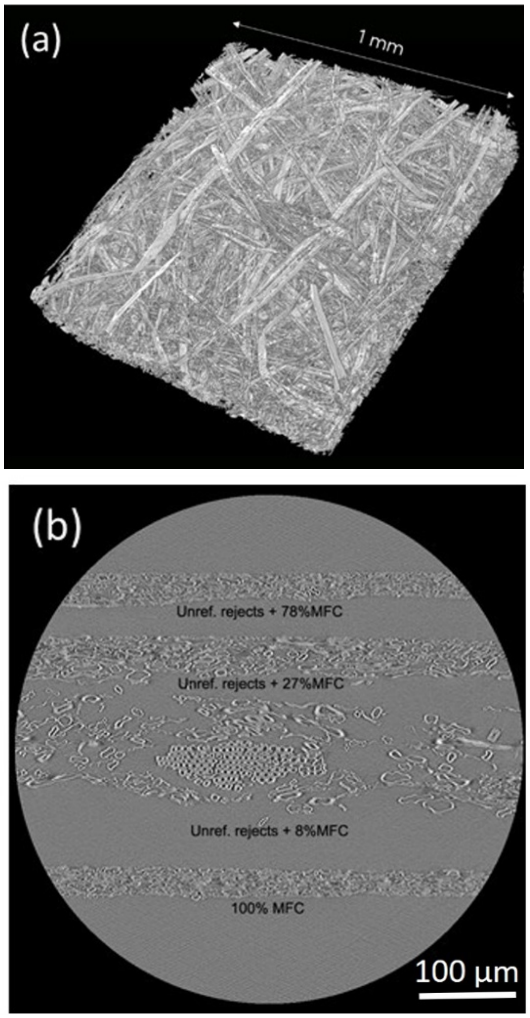

Figure 2 . (a) 3D rendering of a CTMP handsheet (b) Cross section of handsheets with different levels of MFC addition (scanned simultaneously using the multi-sample holder described earlier).

PROJECT 3.2

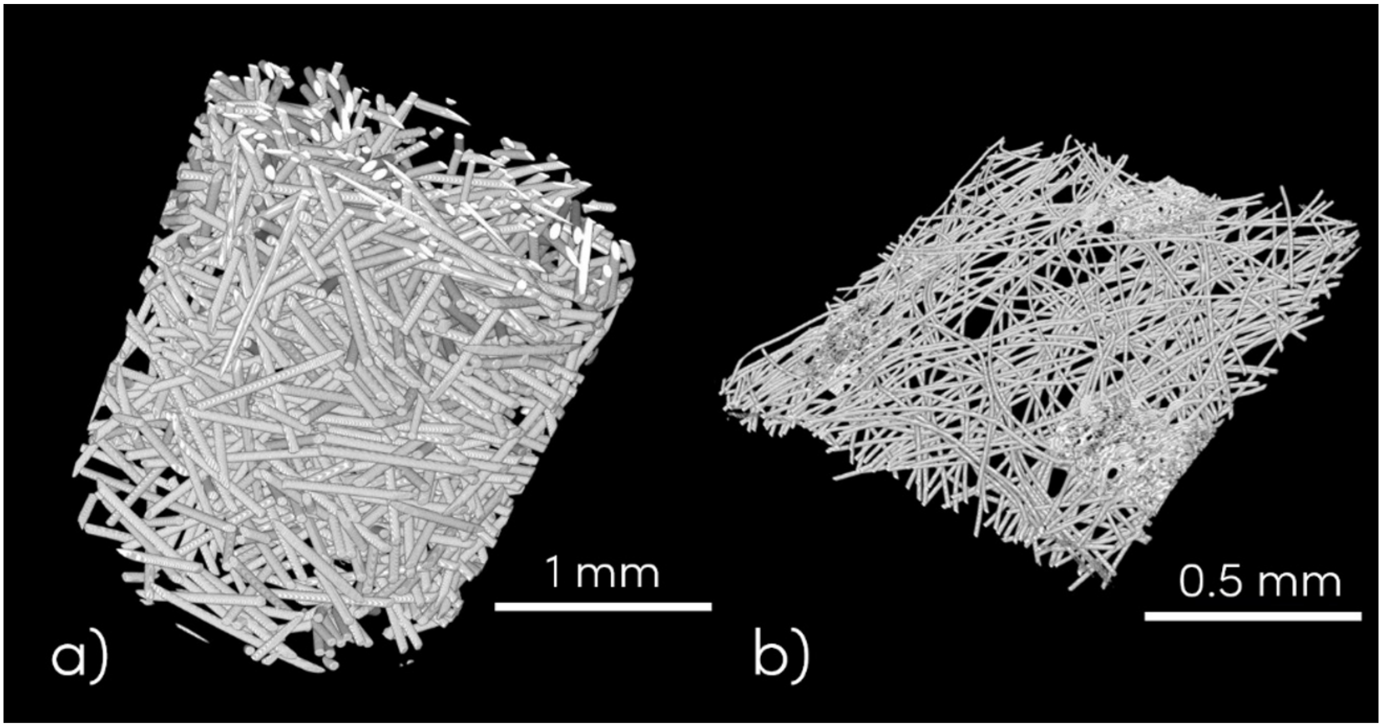

Figure 3. (a) Nylon fibre bundle (fibre length ~1mm) (b) Surgical mask with continuous fibres

The third step was to begin the process of quantifying microstructural features within each 3D-imaged handsheet with the ultimate goal of relating microstructure to macroscopic properties such as tensile strength. Image quantification of paper handsheets is a difficult challenge since artifacts, including partial volume effect and noise, and the inherent complexity of the architecture all hinder reliable use of automatic image analysis algorithms. Within the literature, only a few semi-automatic approaches have been developed for the segmentation of networks made of heterogeneous wood fibres [2-4]. Previously, we presented an automated lumen tracking algorithm to segment selected fibres in the sheet in 3D [5]. Over the past six months we have re-visited this work and extended it to estimate the number of contacts per fiber, as this is a key metric which governs paper strength. Thus far, we have benchmarked this new code's performance against idealized fibre networks. A representative image of a nylon-fibre bundle is shown in Figure 3a where we estimate that, on average, there are about 7 contacts per fibre. We then extended this algorithm to non-woven samples, i.e., a surgical mask as shown in Figure 3b. Over the next reporting period we will extend this algorithm to mechanical pulp hand sheets.

Project 2 – Imaging of Wood Chips

The goal of this project is to understand where bulk is lost during the mechanical pulping process. We are developing protocols for the study of the material's states through the transformation process: from raw materials (wood chips) to treatments to the final product (paper handsheets). Previous studies in the lab aimed to characterize the "bulk potential" of a papermaking furnish by comparing collapsed fibres in a paper sample (using the in-house fibre segmentation algorithm) with fibres of the original wood chips. This showed the potential for evaluating loss in bulk during the mechanical pulping process.

Over the reporting period, aspen wood chips were treated with either sodium sulfite (0.4 M pH10, or, 1 M pH12), alkaline peroxide (pH 13) or water (control) and then imaged in 3D via X-ray tomography at a resolution of 0.7 µm (see Figure 4a). We have also developed an imaging protocol to evaluate the change in structure at different levels of compression. Representative images of the same wood chip before and after compression at 20% strain are shown in Figure 4b. Over the next reporting period we will perform a systematic study to examine the deformation of wood chips under different loading conditions.

PROJECT 3.2

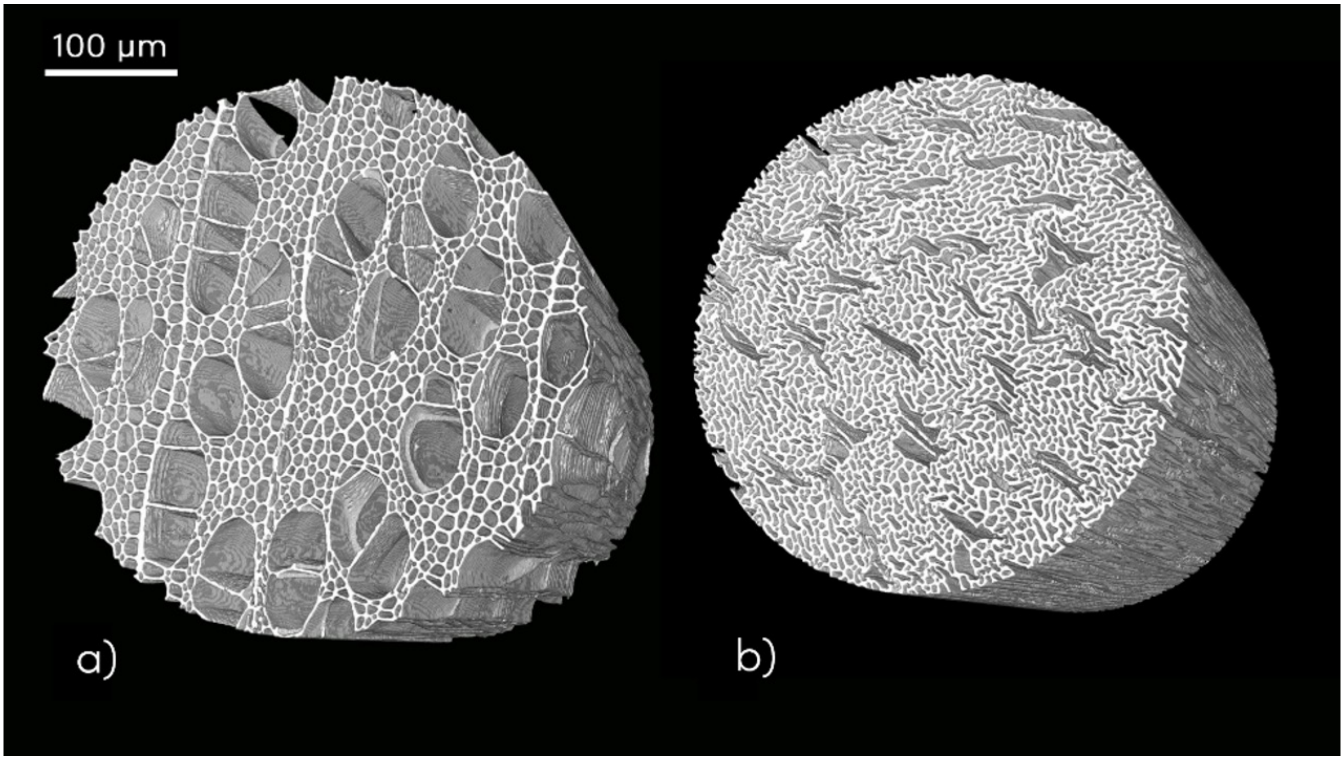

Figure 4. Aspen wood chip scanned (a) before compression and (b) after compression and several days of relaxation. The resolution is 0.7 µm for a field of view of 700 µm. About 3 hours is needed to achieve each scan. It can be observed that a high compression level resulted in a radical change of the wood morphology as the microstructure was heavily deformed.

Future research

Over the past six months, we have developed reliable tools and skills for 3D imaging, and for microstructural quantification of the resulting 3D datasets. Over the next reporting period, we will perform systematic studies to visualize the changes in the structure of paper and wood samples subjected to different process treatments.

PROJECT 3.2

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ERMP PERSONNEL UPDATES

Farewells

Recently the ERMP group has said farewell to graduate student Bryan Bohn who has successfully completed his M.Sc degree. We thank Bryan for all his hard work and wish him the best in all his future endeavors.

Welcoming new arrivals

With the start of this new phase, we continue welcoming several new team members.

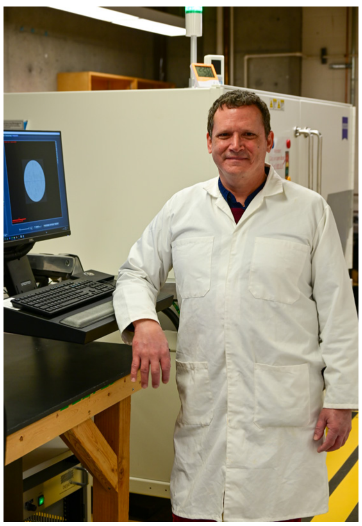
Oskar Ray Gustafasson

Emily Cranston and Emil Gustafasson are happy to introduce the youngest member of the team, Oskar Ray Gustafasson. Oskar was born Oct. 20, 2020 and 6 months has flown by! He is a goofball, always laughing, loves to eat and recently started crawling backwards at great speed. Oskar is lucky to have his father on parental leave with him, now that Mom has returned to work – he's learning Swedish (special thanks to Mark Martinez's gift of Swedish kids books) and spending the long sunny days at all the playgrounds around UBC.

New Staff Members

James Drummond

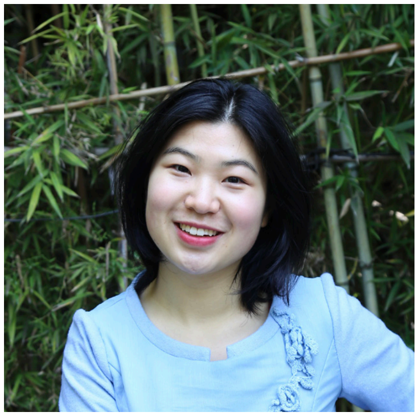
James joined the team in mid-November 2020 as our microscopy technician. He has a B.Sc in Wood Science from UBC, and brings years of experience as a pulp and paper microscopist with FPInnovations and Pulp and Paper Research Institute of Canada, applying light and scanning electron microscopy to the imaging and analysis of wood, fibre, paper, board, bio-nanomaterials and composites. His primary responsibility will be operation of the X-ray tomograph and tomography protocol development, as well as overseeing and applying the other microscopical/imaging equipment in the lab.



Graduate students

Siawei Chen - Project 2.1

Siwei received her Bachelor of Science in Wood Products Processing from UBC in 2021. With years of experience being a research assistant in the Advanced Renewable Materials Lab, she has pursued further graduate studies here. Equipped with the essential lab knowledge and skills, Siwei joined the ERMP team early May and is working under the supervision of Prof. Scott Renneckar and Postdoc Liyang Liu. She has assisted with projects such as the synthesis of lignin-based polyurethane foam and bioplastics from wood-based materials.



Mariana Frias de Albuquerque - Project 2.3

Mariana joined the ERMP program in January 2021 as a PhD student at UBC. She will work under the supervision of Professors Heather Trajano, Boris Stoeber and James Olson. Mariana will study how the specific refining energy and properties of microfibrillated cellulose (MFC) produced from mechanical pulps can be manipulated through enzymatic pretreatment and low consistency refining (project 2.3). Previously, Mariana received an MSc degree from Université Laval (Quebec, Canada) in Engineering of wood and bio-based materials; and a BSc in Engineering from the Federal University of Rio de Janeiro (Rio de Janeiro, Brazil).

ERMP PERSONNEL UPDATES

Postdoctoral fellows

Samira Gharekhani - Project 1.1

Dr. Samira Gharekhani joined the ERMP team as a postdoctoral fellow in March 2021. She is working under the supervision of Prof. James Olson, and contributing to ERMP projects 1.1, 2.3, and 3.2. She received her Ph.D. in Mechanical Engineering – Thermofluids - from the University of Malaya in 2016. Her Ph.D. research was focused on the flow characteristics of suspensions containing refined fibers. In 2016, she joined the University of Tehran as a PDF and worked on advanced functional nanomaterials. She then moved to Lakehead University (LU, Canada). As a PDF, she further developed her multidisciplinary research skills through working on biomass valorization. Later, she worked as a sessional lecturer at LU. Her research interests focused on sustainable materials. She is expert in production of novel bioproducts through physicochemical alteration of biomass e.g., nanocrystal cellulose and lignin for energy and environmental applications. She is a recipient of Marie Skłodowska-Curie Actions individual fellowship (H-2020).

Mengqi Fang - Project 1.2

Dr. Mengqi Fang has received her Ph.D. degree in Chemical and Materials Engineering from the University of Alberta in 2020, and she is currently working as a postdoctoral fellow under the supervision of Professor Bhushan Gopaluni and Professor Yankai Cao. Her research interests include data-based process monitoring, fault detection and diagnosis, Bayesian theory and probabilistic graphical model, etc. Her recent works focus on the image processing of the X-ray tomography outputs using deep learning algorithms and the energy optimization of mechanical pulping processes.

Jingqian Chen - Project 2.3

Dr. Jingqian Chen received a Ph.D. in Chemical and Biological Engineering (University of British Columbia) in 2020. Her Ph.D. research focused on softwood hydrolysis kinetic modeling, Kraft pulping and hemicellulose adsorption to pulp fibres. Jingqian obtained a M.Sc.E. in Chemical Engineering (University of Michigan) on biodiesel production from microalgae. Afterwards, she worked as Research Scientist in industry on coal to chemical technology. Her recent work investigates the sulfite pulping and alkaline peroxide treatment of hardwood chips, the micro-fibrillated cellulose production by enzymatic hydrolysis, pulp refining, and the related microscopy analysis.

Rasmita Sahoo - Project 2.3

Dr. Rasmita Sahoo received her Ph.D degree in Physics from the University of Hyderabad, India in 2017 and her work was on rheological studies of liquid crystals with structural and induced topological defects. Later she joined as a postdoc in the University of Granada, Spain (2018-2019) and investigated the rheological and tribological properties of inverse ferrofluids to prepare a smart lubricant.

In the ERMP program under project 2.3, she will analyse how wood fiber processing conditions i.e, mechanical refining and enzyme treatment affect microfibrillated cellulose morphology and how the morphology of suspended fibers responds to shear under rheology techniques.

LAB AND TRIAL UPDATES

Renovations updates

In our last newsletter, we reported on the Bioproducts Institute renovation project at the Pulp and Paper Centre. Construction activities have now been in full swing for the past 6 months making substantial progress and project completion is expected very soon. As a result of the renovations, a major relocation of several pieces of equipment is also occurring throughout the Centre. Everyone is working to re-design their laboratory spaces to accommodate this reshuffling.

Laboratory updates

A new laboratory room, Lab 114, is being created at the Pulp and Paper Centre, taking over a space that had been previously underused as a storage/workshop area. The development of the lab will house many of the relocated apparatuses and individual research projects moved from the construction activity. This work is still in progress as the equipment is being re-assembled and the necessary infrastructure elements are brought in. However, great strides have already been made to bring the space to full functionality and some research activity has already begun.

The papermaking lab is also undergoing a bit of a rearrangement as well, taking in supplies and equipment from the renovation reshuffling. In this process, a few previously underutilized pieces of equipment are being reassessed and given a second life. Recently this has applied to the Parker Print-Surf roughness tester and Valley Beater. Both have received servicing and re-entered active use after years of inactivity.

Parker Print-Surf roughness tester

Trial updates

The renovation work and the limitations in the number of permitted on-campus personnel have restricted pilot plant activity. However, a few experiments in screening and refining have gone forward in the past few months. Project 2.3 completed a 3rd trial in the investigation of short fibre MFC production, a pressure screen fractionation trial has been completed for industry partner Canfor and a series of pressure screening operations were completed for a research student to investigate flow behavior. In the coming months, the use of the pilot plant is expected to increase with several projects and many trials already lined up.

Pilot plant refining trial with George Soong and Reanna Seifert. Image taken in November 2020.

Valley Beater

PUBLICATIONS

We are pleased to announce the details of the MASc thesis of Bryan Bohn and a recent article published by PhD student, Matthias Aigner.

Theses

Bohn, Bryan, MASc Thesis “A machine learning approach to classification of gas entrainment and impeller wear in centrifugal pumps”, the University of British Columbia, 2021.

http://hdl.handle.net/2429/77247

Journal Articles

Aigner, Matthias, Olson, James and Wild, Peter. "Measurement and interpretation of spatially registered bar-forces in LC refining" Nordic Pulp & Paper Research Journal, vol. 35, no. 4, 2020, pp. 600-610

Upcoming Event

Our next ERMP Steering Committee Meeting will be held in June 3rd, 2021 from 8 am to 12 pm PST through the Zoom platflorm.

All ERMP Steering Committee members will be notified soon with the meeting details. We hope to see you there!

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