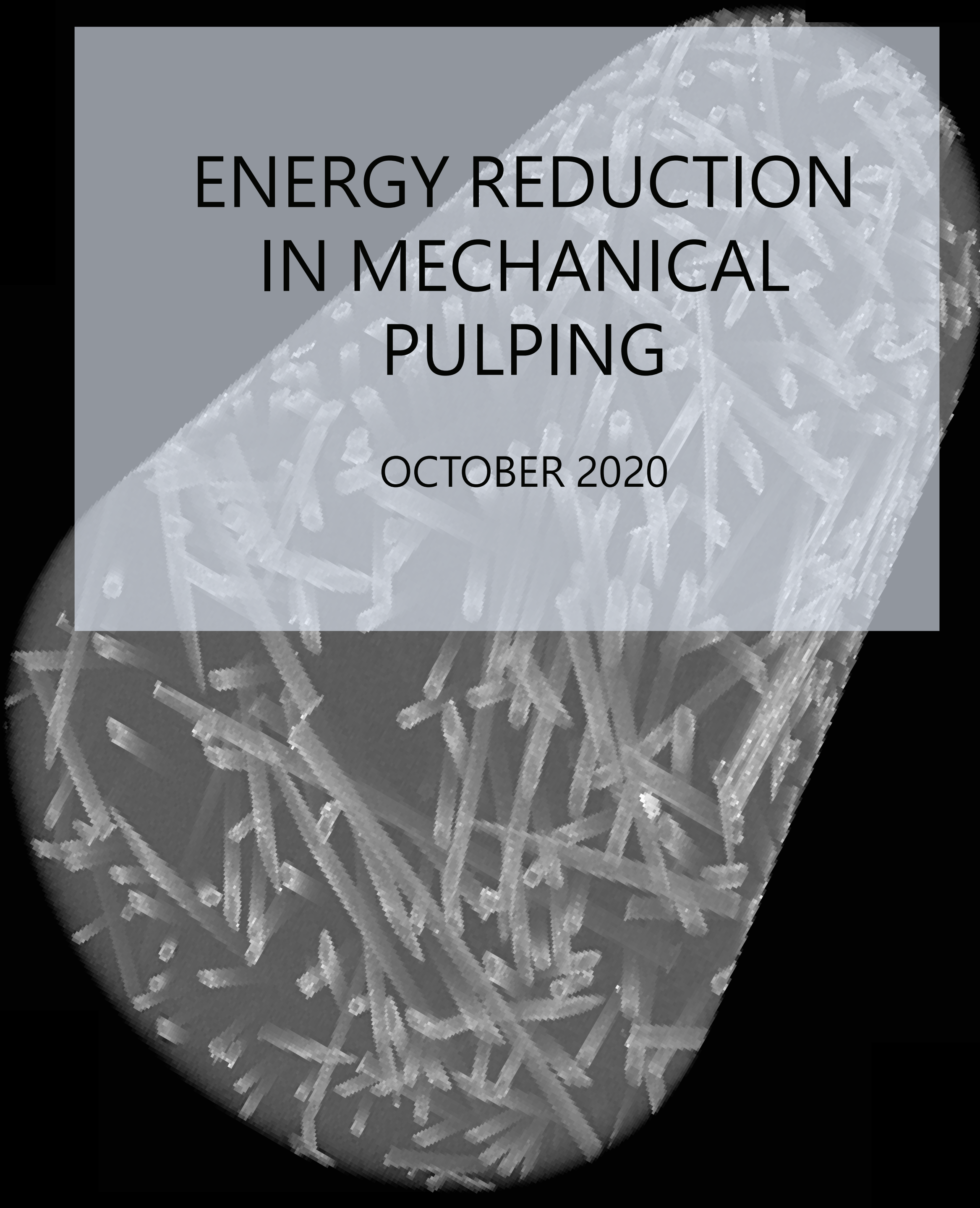
ENERGY REDUCTION IN MECHANICAL PULPING

OCTOBER 2020



THE UNIVERSITY OF BRITISH COLUMBIA

WELCOME MESSAGE

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

I am pleased to announce the start of this third phase of the ERMP program, with an increased scope organized into three research programs with eight projects in total that reflect the need to expand the markets for mechanical pulp. The $3M new cycle of this globally unique partnership was made possible by all of your financial and in-kind commitments. Thanks again for your continued support, we are very optimistic about this new phase starting.

Since our last newsletter, we held a successful Steering Committee meeting at Whistler on June 6, 2019, where several students presented their project updates. Hui Tian successfully submitted her doctoral thesis and successfully defended her Ph.D. earlier this year. Matthias Aigner and Bryan Bohn continue completing their projects and working towards graduation. I invite you to read more about our project updates and publications on the following pages. Considering ongoing traveling restrictions due to covid-19, our next Steering Committee meeting will be held online on November 6th through the Zoom platform. I hope you all have marked your calendars and will join us for this event.

With the start of this new phase, we also have several personnel updates. Daniela Vargas Figueroa has joined the program in July 2020 as the new ERMP Program Manager, while Meaghan Miller had begun a new role as Health Research Development Officer at UBC's Support Programs to Advance Research Capacity. We thank Meaghan for her leadership and many excellent contributions to ERMP and wish her all the best in her future endeavours. Sudipta Mitra, Aurélien Sibellas, Elisa Ferreira, Liyang Liu, and Pierre Betu Kasangana have joined the program as Post-doctoral fellows working closely with ERMP researchers at the start of phase 3. We welcome two new students to the program, Claire Maulit, MASc student, and Cameron Zheng, a Ph.D. student at UBC. We have also recently hired a new ERMP laboratory technician, Michael Bilek, who will be supporting the on-going research at UBC-PPC. Please see pages 28 and 29 for brief introductions to these new members and their work.

In addition to our own group's efforts, it is a very exciting time for UBC's BioProducts Institute (BPI) which has recently become UBC's newest Global Research Excellence (GREx) Institute. This approval opens up new possibilities to support UBC as one of the leaders in bioproducts research and innovation, helping to develop partnerships and attract investments to continue making essential contributions to Canada's global development in this critical area.

Sincerely,

Mark Martinez

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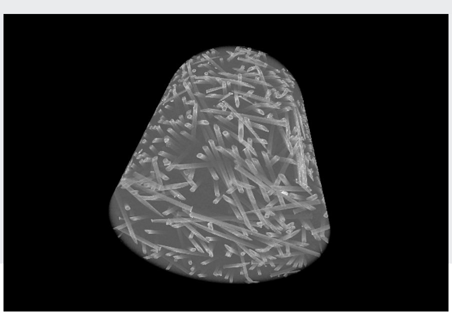

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X-ray tomograph image of a nylon fiber floc taked at UBC - PPC with our new Ziess Xradia 520 Versa. *Photo credit: Professor Mark*

MATTHIAS AIGNER

PROJECT 1.1 - LC REFINER BAR FORCE BASED CONTROL STRATEGIES

Background section

Refiner control strategies are primarily based on global process variables such as rotational speed, flow rate, inlet pulp consistency and outlet fibre length (Luukkonen 2010). In previous work on this project, most recently by Reza Harirforoush, a force sensor was developed to measure shear and normal forces applied by the pulp to the refiner bars to monitor and validate the mechanical interactions in the refiner chamber. This type of sensor has been used in a variety of high consistency (HC) (Olender 2007) and low consistency (LC) refiners (Prairie, 2008, Harirforoush 2016). Most recently, 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 and used in all trials of this project.

Results: Encoder trial UBC

For these trials, a high-resolution rotary encoder (65,536 pulses per revolution) was also installed on the refiner to collect data on the angular position of the rotor. This encoder records the angular position of the drive shaft.

We conducted a set of two trials in 2019 using the refiner plates with bar edge length (BEL) of 2.74. Both trials where conducted at 1200 rpm and a consistency of 3.4%, and the plate gap was varied from 3.5 to 0.2 mm. The first trial used Quesnel River Pulp softwood BCTMP at low freeness and the second trial used Miller Western hardwood aspen pulp.

The position of the rotor bars relative to the stator bars was analyzed in conjunction with the bar force data. Bar force data for the type of plate used typically consists of a repeating sequence of three peaks, each of which corresponds to the passage of a bar on the rotor plate over the force sensor located in the stator plate. The rotary encoder data was used to record these force peaks in combination with the position of the rotor bars as they pass over the sensor. To visualize the passing of the rotor bar over the sensor, Figure 1 shows the bars passing event in incremental steps. This bar passing event is defined from "start" when the leading edge of the rotor bar first overlaps the sensor to the "end" when the trailing edge of the first rotor bar moves off of the sensor.

Figures 2 and 3 show the evolution of the shape of the force plot as the plate gap is reduced for softwood and hardwood fibres respectively. The force plot for large plate gaps shows a single peak, late in the bar passing event. Contrasting that, the force plot for small plate gaps features two peaks. This behaviour is more pronounced in the softwood trials. The shape of the force plot for small plate gaps, features a peak early in the bar passing event and a second peak which is located in the bar passing event similar to the peak for the large plate gaps.

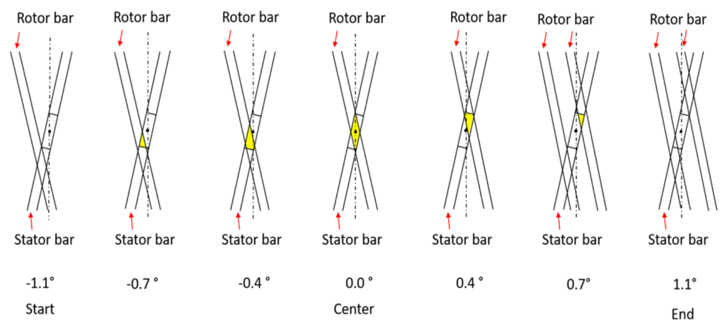

Figure 1. Illustration of the incremental bar passing event at the UBC trial.

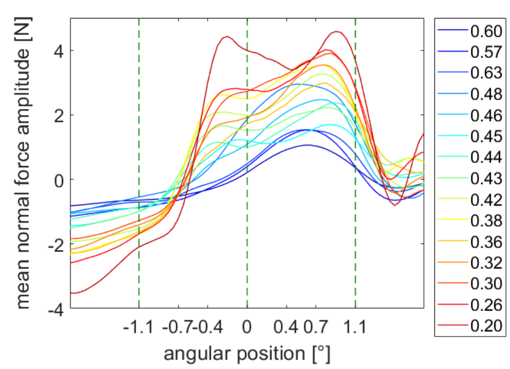

Figure 2. Average normal force profiles for a single BPE for softwood for several plate gaps. Start, centre and end position of the BPE is indicated.

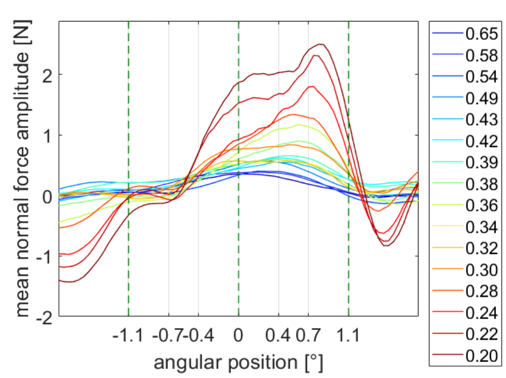

Figure 3. Average normal force profiles for a single BPE for hardwood for several plate gaps. Start, centre and end position of the BPE is indicated.

PROJECT 1.1

The conclusion that we draw here is that the peak early in the bar passing event, which is only significant for small plate gaps, is due to corner force, whereas the peak later in the bar passing event, which is present for all plate gaps, is due to friction force. The corner force arises as the leading edge of the rotor bar approaches the forward edge of the stator bar, compressing pulp in the intervening gap. The friction force on the other hand appears due to the pulp being dragged over the top of the stator bar.

Comparing the changes in the force shape with the fibre length data, we see that the change in the fibre length which we associate with onset of fibre cutting, appears at the same plate gaps at which the appearance of the earlier corner force takes place. This leads to the conclusion that the corner force is the driving force in fibre cutting.

Ongoing research and Results: Catalyst Paper, Crofton

Refiner bar force sensors were installed in the tertiary 56” LC twin disc refiner at the Catalyst paper mill in Crofton, BC, with three sensors being situated in each stator plate at different radial positions. Commissioning of the refiner was in January 2020. The sensors are recording day to day operation and additional trials have been conducted including power curves and bias trials.

In the Crofton trials, no Encoder data was collected, consequently the force shape analysis is conducted in the time domain. In the presented figures, the sample points collected by the DAQ are used as the time domain increment. The rate at which force sample points were collected is 150,000 samples per second. The bar passing event is again defined as from the beginning of the interaction of the rotor bar with the sensor to when the rotor bar leaves the sensor. As there is no position data collected for the Crofton trials, we are not able to determine exactly which force data corresponds to this interval, but we are able to determine the duration of this interaction based on the plate pattern geometry and rotational speed (i.e. 420 rpm). For the sensor closest to the rotor axis (the inner sensor), the middle sensor and the outer sensor, the duration of bar passing event is, on average, 56, 42 and 38 sample intervals, respectively.

Figures 4 to 6 show the shapes of the normal force profile for the inner, middle and outer sensors for bar passing events at different specific refining energies (SRE). The plots for each of the three sensors have a distinct shape which is consistent for all refining energies. The data for the middle sensor features two peaks for the duration of the bar passing event whereas the inner and outer sensors feature only one peak.

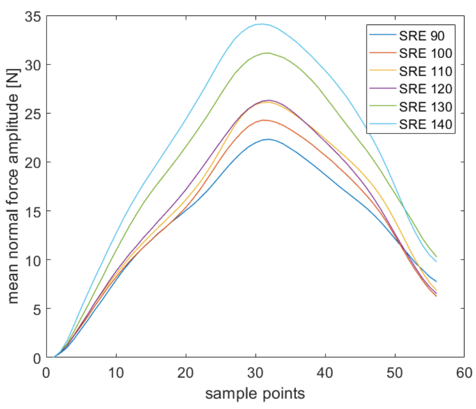

Figure 4. Mean normal force shape analysis at different specific refining energies for the inner sensor.

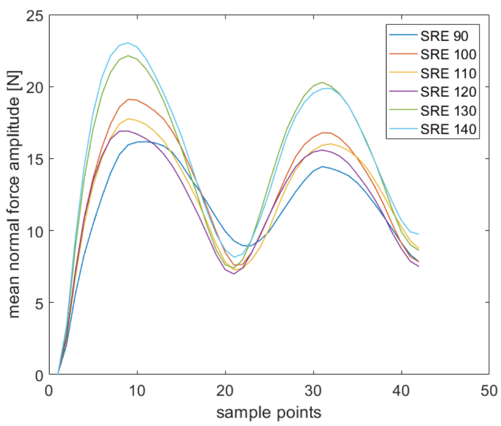

Figure 5. Mean normal force shape analysis at different specific refining energies for the middle sensor.

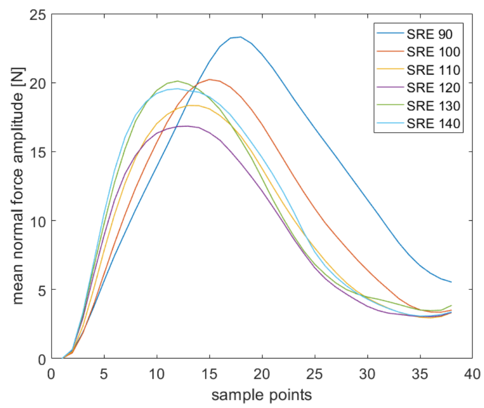

Figure 6. Mean normal force shape analysis at different specific refining energies for the outer sensor.

PROJECT 1.1

Based on the results of the UBC trials, the presence of two peaks in the bar passing event suggests that both corner and friction forces are present at this sensor position. The inner and outer sensors both feature one peak. The peak of the force plot at the inner sensor is located close to the 50% mark of the bar passing event whereas the peak for the outer sensor is located close to the 30% mark. Note that these "locations" are with respect to the valley that precedes the peak rather than with respect to an absolute angular reference point. The location of the force peak "earlier" in the bar passing event for the outer sensor, suggests a more dominant corner force at this radial position. For the inner sensor the location of the force peak is closer to the centre of the bar passing event duration and therefore suggests a closer association with friction force at this sensor position. Under the assumption that corner force is a driving force in fibre cutting the conclusion can be drawn that fibre cutting is either happening solely at the middle sensor or at the middle and outer sensor but not at the inner sensor.

Future work

Further trials at the Crofton mill are planned. This includes trials investigating the balancing of the two refiner sides. In these trials a pressure bias is artificially introduced between the two refiner sides. Force measurements help to improve the understanding of the refiner behaviour in a balanced and unbalanced state. Additionally, arrangements are being made to run test refiner trials at the PPC at 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 the force profile shape.

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PROJECT 1.2 (A)

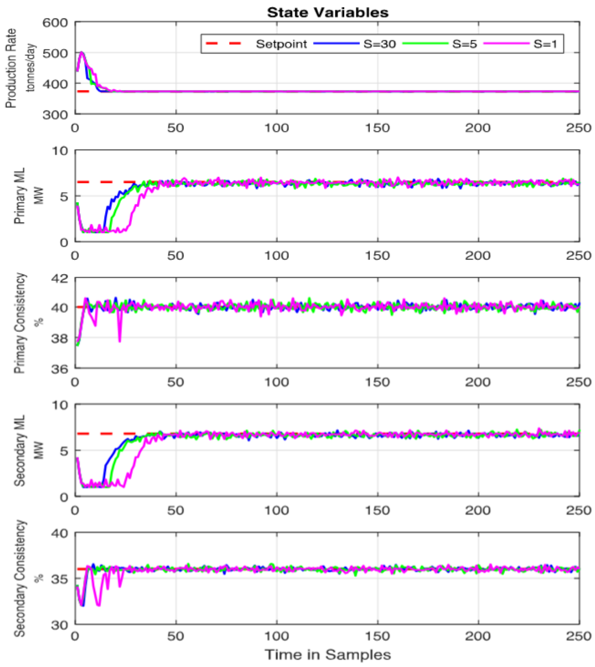
1.2 (A) – ECONOMIC MODEL PREDICTIVE CONTROL OF THERMO-MECHANICAL PULP PROCESSES

Background section

High consistency (HC) mechanical pulping (MP) processes exhibit complex nonlinear dynamics. Model Predictive Controllers (MPC) are often used for optimal operation of these processes. The state-of-the-art commercial MPCs use simple linear models and work well in the neighborhood of previously determined operating conditions. However, they are designed to meet product specifications and other process constraints without accounting for the cost of operating TMP process. We have previously proposed Economic Model Predictive Controller (EMPC) as a means to not only guarantee product quality but also minimize energy consumption. In our more recent work, we have improved the performance of EMPC by adding another layer of sophistication in the form of a stochastic objective function – called Stochastic Model Predictive Control (SMPC). Through this approach we are able to more effectively manage unexpected disturbances and model uncertainties without compromising on the overall performance of the controller.

A major hurdle to online implementation of SMPC is computational tractability. Tractability issues arise as the control formulation involves expected values of the objective function and constraints (which are random variables). For instance, in the most basic SMPC formulation, we seek to minimize the expected value of the objective function (this involves a high-dimensional integral). As a result, we need to approximate the expectation using simulation algorithms such as Monte Carlo sampling or Polynomial Chaos Expansions. These algorithms convert the infinite-dimensional SMPC problem into a standard (finite-dimensional) optimization problem that can be handled using off-the-shelf solvers. Unfortunately, the resulting optimization problems are often computationally expensive. These computational tractability issues are exacerbated in more sophisticated SMPC formulations in which one might seek to optimize complex statistical measures (e.g., variance, conditional value at risk, quantiles, medians). Similar tractability issues arise when dealing with constraints; specifically, constraints for SMPC are often enforced using statistical measures such as chance constraints, quantiles, risk measures, and almost-surely constraints (i.e., constraints are satisfied for all scenarios).

In our recent work, we explored tractable approximations for SMPC. Under the proposed paradigm, we use sample scenarios to transform statistical measures for the objective and constraints into finite-dimensional representations. To deal with tractability issues of the resulting optimization problems, we proposed an approximation technique that is inspired by the quantile scenario analysis method. In our approach, we solve multiple MPC problems for different scenarios to obtain a set of candidate control policies. The observation is that these policies can be computed quickly and in parallel as they do not involve statistical measures. The set of computed control policies forms a candidate set from which we select the policy that best approximates the SMPC solution. This approach allows us to handle complex measures and allows us to prioritize conflicting objectives such as economics and stability. We demonstrated our algorithm using a stochastic version of economic MPC applied to a mechanical pulping (MP) process. The adjacent figure shows the simulation responses considering S number of uncertain scenarios.



PROJECT 1.2 (A)

We are currently having conversations with ANC and Valmet to identify the challenges associated with implementation of our approach. There are four primary challenges: (1) In the simulations, a nonlinear model was proposed to better capture the dynamic behavior of the MP process, while linear models are utilized in current pulp mill control systems. In order to implement the nonlinear model in the existing control systems, the corresponding process model involved in the MPC implementation section needs to be modified. It should be noted that the nonlinear model will induce higher computational burden and possible delays in sending control signals. (2) There are differences between the control structure used in our simulations and the one that is being used at the ANC mill. (3) There are also significant differences between the objective functions used in our simulations and those feasible in the commercial Valmet MPC that is under operation at the ANC mill. Over the next few months, in consultation with ANC and Valmet we are hoping to test our algorithms at the mill.

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BRYAN BOHN

1.2 (B) – ADVANCED PUMP-PERFORMANCE-MONITORING SENSOR SYSTEM

Centrifugal pumps are an integral part of fluid-moving processes around the world. Studies have shown that they can account for 20-60% of expended motor energy in some industries (Pemberton and Bachmann 2010). In 2001, it was demonstrated that centrifugal pumps were the largest singular consumer of industrial electrical energy in the European Union (European Commission 2001). The scope and scale of their usage underscores the potentially global impact that minor improvements in pump performance can have. This project aims to develop improved monitoring methods for quantifying phenomena that reduce the efficiency and longevity of centrifugal pumps.

Measuring Performance

"Performance" is considered as the conditions and phenomena that reflect the overall health of a centrifugal pump. Commercial performance monitoring systems typically fall into three groups; conventional sensors for process measurement, vibration analysis tools, and efficiency monitoring systems.

Each method has shortcomings. Conventional sensors are the simplest and most accessible, providing basic information about pressure, temperature, flow, and other real-time parameters of an operating pump. However, they are also the most rudimentary, leaving the onus of interpretation entirely to the operator.

Vibration measurement systems use accelerometers to evaluate structural vibrations to identify adverse conditions, such as bearing faults and cavitation (Abdulaziz and Kotb 2017). Yet, like conventional process measurement, interpreting the spectra of vibration frequencies typically falls to manual interpretation. More critically, subtle conditions that do not transmit strongly into the pump structure may be difficult to detect and persist unnoticed (Jami and Heyns 2018).

Efficiency monitoring uses multiple sensors to calculate the proportion of input power being converted into productive fluid work, which can then be compared to the best efficiency point (BEP) defined in the manufacturer's pump performance curve chart. However, in the presence of impeller wear, adverse fluid conditions, multi-phase flow, or unknown pump modification, the accuracy of the performance curve chart can be diminished, leaving the pump without a viable reference for what "good" efficiency should actually be.

The shortcomings of existing methods imply a pressing need for more affordable, flexible, accessible methods for monitoring the health of centrifugal pumps.

Summary of Research

Previously, a magnetic sensor was developed to measure the extent of mechanical wear from the backing-plate edge of an impeller (Khoie et al. 2015; Bohn, Khoie, et al. 2019). A multi-sensor approach using machine learning to characterize adverse conditions was also proposed (Bohn, Olson, et al. 2019). Current work focuses on a method that uses the dynamic measurements from a solitary pressure sensor at the pump discharge to classify undesirable operating conditions. The objective is to quantify complex phenomena using only conventional, low-cost sensors that would be accessible to any centrifugal pump operator.

Since the last newsletter update, the system has been simplified to employ a single pressure transducer at the pump discharge, rather than the multi-sensor method initially envisioned. This was motivated by preliminary experiments, which suggested that the additional presence of an accelerometer, intake pressure transducer, and efficiency determination instrumentation was redundant for the testbed pump, and that sufficient classification of adverse operating conditions could be attained using a simpler configuration. In the proceeding months, training data sets have been compiled for the adverse phenomena and classification has been successfully demonstrated.

The method is evaluated on two distinct conditions; radial material erosion from the tips of the pump's impeller and distributed gas entrainment (i.e. bubbles) in the working fluid. These conditions are selected based on their prevalence, performance impact, and difficulty to characterize using existing monitoring methods. The approach is validated using both simulated and experimental data. Each phenomenon is evaluated in its own study, but using the same classification method.

Technical Approach

The severity of radial impeller erosion and air entrainment is determined using characteristic fluctuations in a pump's discharge flow. The fluctuations in the raw pressure transducer signal are decomposed into statistical measures, called features.

PROJECT 1.2 (B)

These features are then used as the inputs to train a multi-layer perceptron (MLP), which is a type of artificial neural network (ANN). After training, the ANN is suitably configured to identify the presence of the phenomena of interest for the given pump.

In the impeller wear study, the severity of the tip erosion is defined by impeller loss ratio α_{imp}. The ratio is determined by the actual impeller's operating diameter D_w and that of the original, unworn impeller D_o, as shown in (1).

$$\alpha_{imp} = \frac{D_o - D_w}{D_o} \quad (1)$$

α_{imp} denotes the relative reduction in impeller diameter from its unworn state and is discussed in the percentage form. $\alpha_{imp}=0$ corresponds to an unworn state. Loss ratios up to $\alpha_{imp}=3\%$ are evaluated.

In the gas entrainment study, the gas and liquid constituents of the working fluid are water and air, respectively. The severity of gas entrainment is characterized by the dimensionless volume fraction of air φ_{air}, which is defined by the ratio of the average volume of air v_{air} and average volume of water v_{water} passing through the pump over time.

$$\varphi_{air} = \frac{v_{air}}{v_{water}} \quad (2)$$

To first establish the dynamic pressure effects of each phenomenon, a model of a simplified centrifugal pump is simulated using COMSOL Multiphysics software. The model is shown in Figure 1.

The size and geometric complexity of the model pump are kept to a minimum to reduce the computational load over the time-dependent study. A counter-clockwise rotating frequency ω is applied to the impeller. The discharge and intake are bounded by fully developed flow conditions with an average pressure and average velocity, respectively. Turbulence is modeled using the Realizable k-ε method. The air-water fluid interface is calculated using the level-set model.

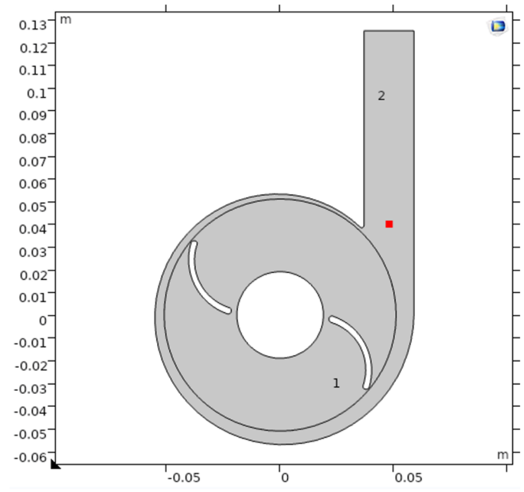

Figure 1. A 2D simulation of a centrifugal pump with a two-blade, 102 mm diameter impeller. The location of the pressure probe is indicated by the red dot. The interior circle corresponds to the impeller's diameter.

Dynamic pressure measurements are collected at the pump discharge, near the mouth of the volute. Simulated pressure measurements for a healthy condition are contrasted with severe air-entrainment ($\varphi_{air}=8\%$) and wear ($\alpha_{imp}=3\%$) conditions in Figure 2.

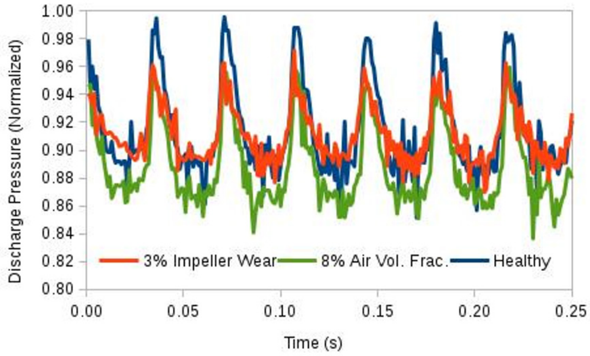

Figure 2. A 0.25 s sample of simulated discharge pressure signals for a pump rotating at 13.75 Hz. Each sample is normalized.

The signals show distinctions between the healthy and unhealthy states for both phenomenon. To quantify these variations, each pressure signal is broken down into an array of statistical values, including variance, skewness, kurtosis, energy, autocorrelation energy, and other frequency-domain measures. These values are normalized and assembled into a list called a feature vector, which is a simplified representation of each pressure sample. Taking samples across many impeller speeds and conditions, from healthy to severe states, yields a spread of data that can be used for machine learning.

PROJECT 1.2 (B)

Plotting a particular feature shows its relationship to the adverse phenomenon. For example, Figures 3 shows that the discharge pressure signal variance decreases as impeller wear worsens. Here, the influence of impeller rotating speed has been normalized out of the sample.

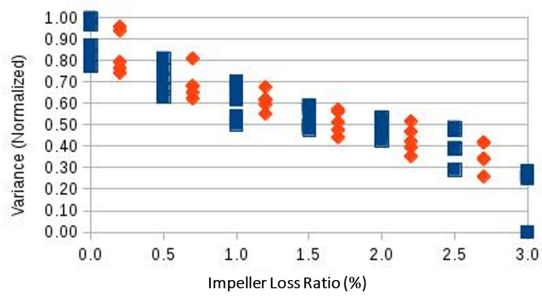

Figure 3. Variance of the simulated discharge pressure signals as a function of impeller loss ratio. The blue and red symbols indicate whether the data point was used for training or testing of the MLP, respectively.

Classification is done using a MLP with one hidden layer containing four nodes, as shown in Figure 4.

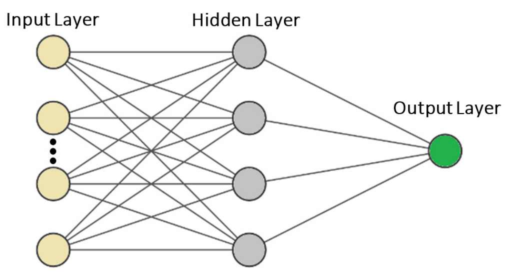

Figure 4. The architecture of the implemented multi-layer-perceptron (Lenail 2019). Each node in the input layer corresponds to a statistical value in the feature vector for a given sample.

The extent of impeller wear or gas entrainment in a given sample is identified using a binary tag 0 or 1, corresponding to an acceptable or unacceptable operating condition. The thresholds for acceptable amounts of impeller erosion or material loss can be changed to suit a particular centrifugal pump.

Classification Performance

70 states are simulated across varying impeller wear and rotating frequencies. The samples are split 50-50 into training and testing sets. Using this configuration, after $4{\cdot}10^4$ training iterations, the MLP achieves a classification success rate of 91% for identifying impeller wear states exceeding 1.5%. Applying the same method and MLP to classify a set of 150 experimental pressure measurements of varying air entrainment and rotating frequencies yields a classification success rate of 89% for air volume fractions exceeding 2%.

Summary and Future Work

A novel method for classifying impeller erosion and gas entrainment has been demonstrated. The technique employs a multi-layer perceptron to interpret characteristic fluctuations in signals from an ordinary pressure transducer at the pump discharge.

In practice, the neural network would be trained by the pump manufacturer by amassing a data set of dynamic discharge pressure measurements in many conditions. The configured classification tool would then be provided to the pump user, who would apply it to test their own pressure measurements for adverse conditions. The method is envisioned to be expanded to detect detrimental conditions beyond impeller wear and gas entrainment. In the future, the characterization will be refined to demonstrate multi-class and continuous-value regression classification for both phenomenon.

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PROJECT 1.3

1.3 – CREATING LOW ENERGY SHIVE-FREE PULPS

Background section

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.

This project builds on previous work on the use of chemicals/enzymes combined with low consistency refining aimed at the electrical energy reduction in the production of mechanical pulp (Chang et al 2010, Chang et al 2016). We have shown that highly alkaline peroxide treatment can reduce the energy required to obtain a given tensile strength by 300 kWh/t. However, the high alkalinity resulted in less than optimum brightness gain. Introduction of a two-stage process allowed optimization of the first stage for brightness gain and optimization of the second stage for strength development. 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, we explored the effect of recycling the alkaline second stage liquor for use in the first stage with the aim of cost reduction.

Experimental

Figure 1 shows the experimental plan using a low freeness spruce thermomechanical pulp (TMP). A typical first stage peroxide bleaching process was followed by addition of extra caustic in a second lower temperature stage. There was no wash between the two stages. One set of experiments was conducted with no liquor recycle, the other with recycle of the spent liquor from the second stage with additional alkali added to the first stage liquor to bring the alkali charge in the second stage to 2.55% or 4.05%, the same as used in the second stage without recycle. The alkali charge in the first stage was 1.95% bringing the total alkali charge with or without recycle, to 4.5% and 6% respectively.

Results

As shown in Figure 2, a single stage brightening process increased the tensile strength by 5.4 N.m/g. The second stage alkali treatment at a total alkali charge of 4.5% increased strength by a further 10.3 N.m/g. The same strength could be achieved with alkali recycle with a savings of 21% in total alkali charge. Use of 6% total alkali charge showed no benefit in further tensile gains.

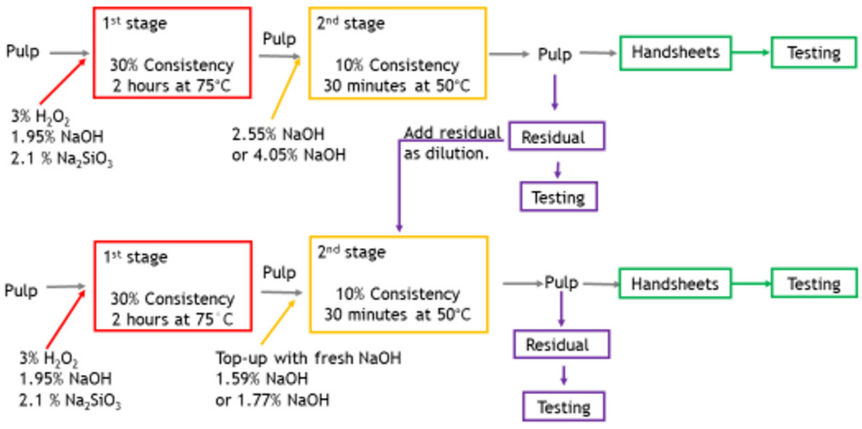

Figure 1. Experimental sequence for two-stage high alkalinity peroxide treatment, with and without liquor recycle.

PROJECT 1.3

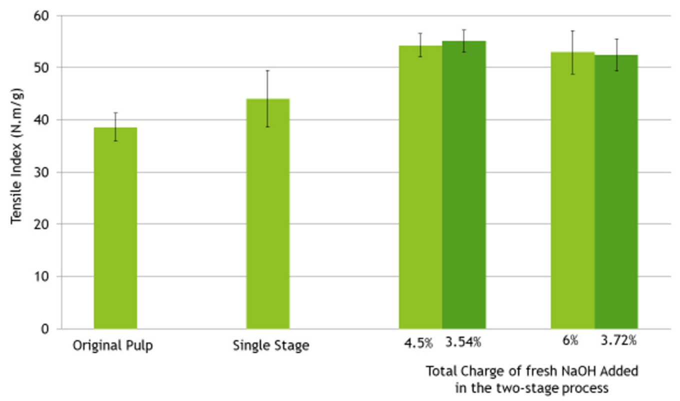

Figure 2. The effect of liquor recycle on tensile strength. Dark green indicates liquor recycle.

Figure 3, shows the impact of recycling alkali on pulp brightness. The single stage process yields the highest brightness of 72.2% ISO as expected from previous work. There is a penalty of 5.3 points in brightess in the second alkaline stage. Interestingly liquor recycle not only reduces the required total alkali charge by 21% but seems to have a benefit in reducing the brightness drop. At the total charge of 4.5% alkali with liquor recylce, the brightness drop was limited to 1.5 points. The reason for this is not clear as no residual peroxide was detected in the recycled liquor. Again increasing the total alkali charge to 6% showed no benefit.

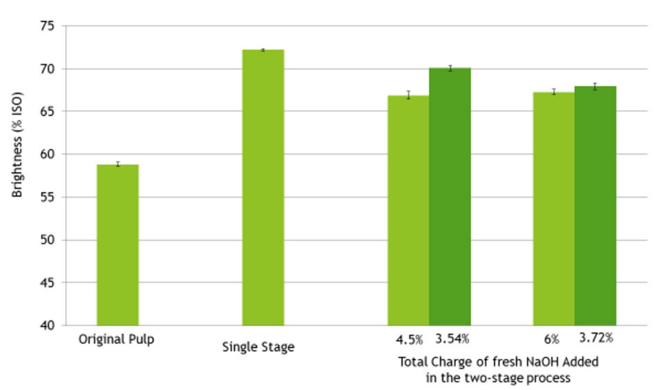

Figure 3. The effect of liquor recycle on brightness. Dark green indicates liquor recycle.

Conclusion

In two stage highly alkaline peroxide treatment of mechanical pulp, recycle of the alkaline liquor from the second stage, maintians the tensile gains and reduces brightness loss while saving 21.5% in alkali charge.

Future research

The response of softwood TMP to highly alkaline peroxide is well documented. A fundamental understanding of the mechanism of the strength development is lacking. Also, prior to mill trials further work needs to be done on the impact of changing process variables on strength development. Future work in this area will addresses these issues.

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

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PROF. SCOTT RENNECKAR

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

Background section

What: This is a new project to investigate valorization of thermomechanical pulp (TMP) fines by changing their surface chemistry using sustainable and scalable methods. Fines will be made to become much more hydrophobic or extremely hydrophilic through chemical modification. In so doing, the hydrophobic fines will be evaluated for potential to serve in new markets such as additives in plastic composite applications, while the hydrophilic fines will be evaluated as a synergistic material with other wet-end additives to enhance handsheet properties leading to handsheet improvement.

TMP fines, particles that pass through a 200 mesh wire with less than 75 micron particle dimension, compose up to 30% by weight of TMP (Sundberg et al 2003). These fines are either loss through the papermaking process or can interfere with paper properties dependent upon their concentrations. In a nut shell, fines are "specks" of wood fiber that either consist of an entire box-like wood cell called a ray cell, or arise from cell fragmentation (Odabas et al 2016). The chemical compositional analysis of these materials revealed higher lignin concentrations than the bulk of the fiber, in the range of 35-40% dependent upon fine geometry (Odabas et al. 2016). Further, extractive content was enriched on the surface of the fines compared to the bulk wood (Kleen et al. 2003). Hence these materials are small and heterogeneous, and can negatively impact sheet properties if too many fines are included in the final sheet composition (Page 1969). As such, the project aims to modify these fines in a way that accounts for this heterogeneity and utilize them in applications where their small size is a benefit to the material.

How: The goal is to make the modification of fines efficient by utilizing green chemistry principles, while at the same time understanding the role of the various wood fibre components on modification. This latter step will involve selective delignification and modification of the isolated lignin and the residual fine substrate, independently. Further, over the course of the project by utilizing a dynamic draining jar, we will systematically evaluate the modification of both flake and fibrillar geometries. These isolated components will be modified with the chemical approaches described next.

Recently, Liu et al, reported on the direct esterification of technical lignin utilizing organic acid such as propionic acid (2018). These acids selectively modify available aliphatic hydroxyl groups, converting them into esters. Different acids, such as octanoic or oleic acid with various carbon chain lengths can change the hydrophobicity of the lignin. Further, certain organic acids are quite benign and even can be sourced from softwood kraft mills making them an abundant source if they can be properly utilized. Significant degrees of substitution for technical lignin can occur based on the reaction conditions even with long chain fatty acids (Liu et al. 2019). We plan to use three different acids with hydrocarbon chains of different carbon length (C3, C8, and C18) to establish modification that has different degrees of hydrophobicity for our different fine substrates. Acid catalysts will be placed in the reaction mixture to facilitate Fischer esterification of the fibre; however, this can have some negative impacts on the molecular weight of the polysaccharides and thus will be closely monitored. Overall, heterogeneous esterification with different acids would allow the fine surface to be tuned to interact with hydrophobic matrices offering a path towards combining these into melt processable bioplastics.

Further, modification of heterogenous substrates like cellulose fiber (Nagaoka et al. 2005) or isolated components (Wang et al. 2017) have been reported with cyclic anhydrides. The interesting approach to utilizing these reagents like succinic or glutaric anhydride is that the corresponding ester derivatives have free carboxylic acid groups. When placed in water these acids can deprotonate and provide highly charge surfaces impacting the wettability and bonding capacity of the fines. Wang et al. found that when utilizing glutaric anhydride as a modifying agent, lignin was more reactive than hemicellulose and cellulose from bagasse substrates (2017). With the high lignin content in the fines, this approach seems appropriate to modify the cells as there is little waste product. Further, different cyclic anhydrides were used to modify kraft lignin and was found the lignin could be an effective dispersant (Delgado 2018). In summary, there is a high probability of successfully modifying fine surface chemistry in heterogeneous reaction conditions achieving either carboxylated or aliphatic ester derivatives of the fines that would make them hydrophobic or hydrophilic, respectively.

PROJECT 2.1

Why: With their small size, fines may be of interest to make uniform wood additives for applications in polymer fillers, such as for 3-D printing inks, coatings, and enhanced additives for handsheet-- if the chemistry at the surface can be tailored for specific applications. An example application for fines may be found in additive manufacturing. Fused deposition modelling inks for 3-D printing require exceptionally refined wood fiber, below 250 microns in diameter, which was reported to still cause issues with nozzle blockage during printing (Wimmer et al. 2015). Hence, fine fractions which are smaller than 75 microns may be appropriate additives for these materials, as the energy has already been used to make such a small sized particle. The trick, once isolated, is getting the fibre properly dispersed in a matrix. Surface energy plays a large role in dispersion of fiber into hydrophobic (melt processable) matrices and this factor can be controlled through surface modification. After modification, we will evaluate hydrophobic fines for potential to be dispersed in hydrophobic bioplastics and polymers by melt compounding fines at various wood loading with materials such as polylactic acid (PLA). If successful, we will extrude filaments with better shapability and performance to be used in 3-D printed structures. The hydrophilic fines will be added to suspensions of fibre and handsheets will be created as a function of modified fine loading. It is expected that performance of the handsheets will be enhanced with charged fines and may be useful for further modification with mineral additives or cationic polyelectrolytes to better tune the handsheet properties.

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PROJECT 2.2

2.2 – FROM TREES TO TREATMENT FUNCTIONALIZING TMP EXTRACTIVES

Background

Canada's forests are rich in extractives such as terpenes and resin acids. In the mill environment, these extractives create operational challenges for water use, water treatment and fibre product quality. However, extractives can be used directly or readily modified using catalytic technologies to produce more valuable bio-chemicals to meet the global demand for sustainable alternatives to petroleum (Beatson, 2011). Terpenes, such as limonene, pinene, terpinolene and camphene, are the "original" natural solvent, turpentine, and have long been used in cleaning and degreasing applications. More recently, these extractives have been used as food additives, natural pesticides and emulsifying agents in the pharmaceutical industry (Mercier et al., 2009; Panel Report, 1977; Rodrigues- Corrêa et al., 2012). Furthermore, upon catalytic chemical modification, such as hydrogenation, oxidation, esterification, or isomerization these terpene starting materials are being used in the fragrances and flavours industry as well as the pharmaceutical industry. Mechanical pulp mills present a unique opportunity to extract these oils unmodified by chemical pulping liquors.

On average 10-40% of mechanical pulp is fines which may be thin, thread-like particles or bulkier pieces of fiber, cell wall and middle lamella, and ray cells. Mechanical pulp fines are traditionally divided into two classes, "slime" fines which have fibrillar morphology and "flour" fines that have a flake like physical appearance (Brecht and Klemm, 1953). Kangas and Kleen (2004) demonstrated that higher order terpenoids such as abietic acid are concentrated on the surface of fibrillar fines. By capturing and sorting fines through a combination of settling and gel fractionation (Madani et al., 2010; Madani, 2011) it may be possible to generate a concentrated extractive stream suitable for further purification and catalytic upgrading.

Dr. Schafer, a Canada Research Chair in Catalyst Development, a Fellow of the Royal Society of Canada and the author of more than 110 scientific publications on the development of catalysts for green chemical transformations that add functionality to simple feedstock chemicals of limited use. Specifically, Schafer has developed a unique catalyst system that can directly functionalize terpenes that are available in the extractives targeted in this proposal (DiPucchio et al., 2019). Notably, the Schafer group has best-in-class, patent protected, catalytic technology capable of the direct amination of terpenes.

Naturally occurring terpenoid-alkaloids (i.e. compounds with oxygen and amine functional groups) are a known class of natural products that have applications from anti-microbials to anti-cancer agents to drugs for the treatment of heart disease (Cherney et al., 2011). It is proposed that the novel terpenoid-alkaloids generated by Dr. Schafer from the extractives furnished by Dr. Trajano will have applications as potent pharmaceuticals, herbicides, and insecticides.

The objectives of the proposed work are:
1) Characterize and isolate extractives enriched on the surface of fibrillary fines. Models describing desorption of extractives from the solid surface to the fluid phase will be prepared.
2) Establish catalytic protocols for preparing synthetic terpenoid-alkaloid products. Characterize the new products and assess their preliminary biological activity.

Results - Amination of Model Terpenes

Purified limonene or β-pinene (Sigma-Aldrich) were subjected to catalysis with Schafer's $Ta(CH_2SiMe_3)_3Cl_2$ and ureate salt catalytic system. Limonene and β-pinene were selected for this study as they had been previously identified in the filtrate of chip washing systems at Powell River Catalyst. The catalyst features a metal, tantalum, that can sourced from Canadian suppliers, and using inorganic chemistry protocols the catalyst can be readily assembled and delivered to the reaction conditions. Tantalum is a low-toxicity metal, meaning that at the end of the reaction the tantalum oxides can be disposed without special protocols. The catalytic reaction features a transformation where all atoms of the starting material are in the product and no waste is generated, meaning that green chemistry approaches are featured in the functionalization of these oily terpene products.

Recent advances in the Schafer lab have shown that these reactions can also be completed solvent free and improved catalyst formulations can reduce the length of time required and the cost of the transformation (DiPucchio et al., 2018). Upon reaction completion the amine product can be easily purified using a simple washing protocol that results in the purification of the aminated terpene products as solids. Most important to these transformations, the molecular complexity furnished by nature, is not destroyed and the complex architecture, which is key to the selective biological function, is retained.

PROJECT 2.2

These preliminary results illustrate the potential to add functionality to terpenes, a diverse class of structurally complex oily/greasy compounds of low value.

By taking advantage of the bioservices laboratory available within the Department of Chemistry, preliminary in vitro screens for anti-bacterial, anti-fungal and anti-viral activity will be completed. These results would be used to attract potential industrial partners for commercial development. Furthermore, Mr. Zheng will undertake a literature review of medicinal chemistry literature to identify classes of therapeutic molecules that could be assembled using this approach. Previously patented and approved food/drug architectures present lower barrier entries to commercial products.

Future research

Isolation, fractionation and characterization of extractives

The first milestone will be to characterize the extractives adsorbed on fines recovered from partner mills. After compositional analysis of the feedstock is complete, central-composite design of experiments will be used to identify conditions to maximize desorption of extractives from fines. Experimental design will consider: solids consistency, slurry temperature, electrolyte addition (type and concentration), pH and residence time. Solid and liquid samples before and after desorption will be analyzed to ensure mass balance closure is achieved. GC-MS, LC-QqQMS and LC-UV-HRMS will be employed for the compositional analysis of the liquid phase.

The chromatographic data obtained will allow to compare the samples received from the partner mills and to estimate the economic potential of the identified major compounds, notably terpenes and resin acids. The chemometric analysis will be performed using Principal Component Analysis (PCA) to highlight the variability of extractive composition in the samples as a function of the desorption parameters used. The main terpenes or resin acids will be isolated and purified and their chemical structures will be confirmed by NMR spectroscopy.

We anticipate that further concentration will be necessary following desorption of extractives. Liquid-liquid extraction will be investigated; solvents will be selected to harmonise with subsequent catalytic transformation conditions. Mathematical modelling of the evolution of the extractive concentration into the fluid phase will follow a multiphase Eularian-Eularian approach. Closure relationships for the kinetics of the dissolution of terpenes by the solvent will be determined through independent experiment. The model will be used to determine the optimum strategy to maximize yield with minimum solvent.

Amination of Terpenoids

Catalytic protocols will be established for preparing synthetic terpenoid-alkaloid products in one atom-economic transformation. This alkene hydrofunctionalization reaction reacts directly with the naturally occurring C=C bond of terpenoids and adheres to the principles of Green Chemistry and Engineering.

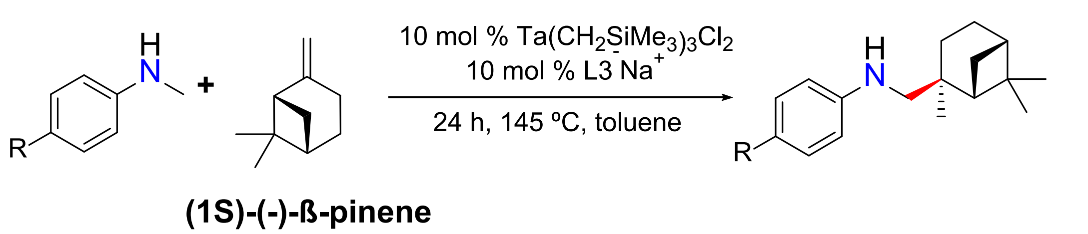

Figure 1. Hydroaminoalkylation of (1S)-(-)-β-pinene with secondary amine.

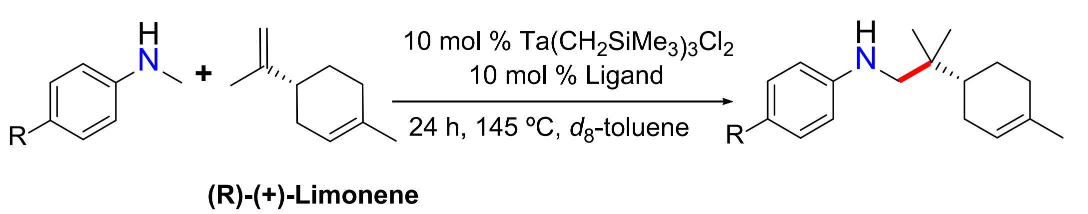

Figure 2. Hydroaminoalkylation of (R)-(+)-limonene with secondary amine.

PROJECT 2.2

Preliminary results demonstrating the viability of this method for application on commercially available monoterpenes, such as β-pinene and limonene has been disclosed (Di Pucchio et al., 2019). The effectiveness of alkene hydrofunctionalization of the larger diterpenoids concentrated on fibrillar fines is unknown. Amination of resin acid streams generated in part 1 will be demonstrated. Operating variables such as amine reagent will be tested. Reaction metrics including diterpenoid conversion and product yield will be assessed.

A range of diversely amine functionalized synthetic terpenoid-alkaloids will be prepared. The new products prepared here will be fully characterized, and their preliminary biological activity will be explored using cell and bacteria in vitro assays in the UBC Biological Services Laboratory in the Department of Chemistry. Promising lead compounds will undergo further evaluation in collaboration with the UBC Centre for Drug Research and Development (CDRD). Here select terpenoid-alkaloid products will be subjected to high throughput screening campaigns against a diverse array of biological targets to identify new lead compounds for drug development efforts. Early results from UBC Biological Services Laboratory and CDRD will be used to identify structure-activity relationships that can guide selection of reactants in future terpenoid-alkaloid synthesis efforts.

An alternative value chain will be explored: the synthesis of renewable polymers from terpenes (Wilbon et al., 2013). These materials are known, but the economic drivers are insufficient to render this class of green polyolefins competitive with petrochemically derived materials. However, we proposed that by adding functionality using catalytic amination technologies available to the Schafer group, functional, responsive polymers can be manufactured for high-value applications such as self-healing binders in battery manufacture, solid electrolytes in solar cell applications and drug delivery materials.

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PROJECT 2.3

2.3 – MFC PRODUCTION, CHARACTERIZATION, AND PROPERTIES OF MECHANICAL

This project aims to produce a low-grade Micro-Fibrillated Cellulose (MFC) from mechanically fractionated fines as the source-material. However, MFC production is generally very energy intensive and therefore costly. The high energy cost MFC production can be mitigated through pre-treatments such as acid hydrolysis or enzyme degradation (Miao & Hamad, 2013). Endoglucanase is the most common enzyme for MFC production; it decreases fibre length, cellulose degree of polymerization, and produces fibres with a larger aspect ratio (Henriksson et al., 2007; Pääkkö et al., 2007; Tian et al., 2017). Xylanase and mannase, which act on hemicellulose, have also been applied in combination with endoglucanase. There is no consensus on the effects of xylanase and mannase treatment: changes in degree of fibrillation and fibre swelling have been reported (Hu et al., 2011; Long et al., 2017; Tian et al., 2017). Recent work at UBC indicates that the order of operation (enzyme treatment, mechanical refining) changes the MFC properties. There is little understanding of how to select and apply enzyme treatments in combination with mechanical refining in order to target specific MFC properties.

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.

Preliminary results

Production of high bulk mechanical pulps was completed by pressure screen fractionation followed by high specific energy refining the short fibre fraction into micro-fibrillated cellulose (MFC) and recombining the MFC with the long fibre fraction. Previous research demonstrates that paper made from fractionated long fibres results in an open, porous, high bulk sheet structure (Olson et al, 2001). Further, MFC generated through high energy refining combined with virgin pulp displays improved bulk, freeness and tensile properties over conventionally refining at an equivalent energy per tonne (Jahangir & Olson, 2019). Expanding upon these results, the current study hypothesizes that the long, coarse mechanical pulp fibre fraction will create an open structure, high-bulk sheet and the introduction of MFC will strengthen the bonds between fibres to provide sufficient tensile strength. The ability to create highly open, porous and bulky sheets with sufficient strength will provide an opportunity for mechanical pulps to enter into new packaging and absorbency products and markets.

Experimental set-up

To create the short fibre MFC the source pulp was fractionated using a Beloit MR8 pressure screen with a 0.8mm smooth-holed screen cylinder. The feed pulp consistency was 1.1%, the volumetric reject ratio was 0.2 and the velocity through the holed apertures was 0.3m/s. The resulting reject thickening factor was 2.75 with a mass reject ratio of 56%. The short fibres (44% of total pulp mass) were subsequently low consistency refined (LCR) on a 16-inch Aikawa refiner with a plate that had a bar width of 1.0 mm, groove width of 1.3mm, and Bar Edge Length of 12.9. The refiner was operated at an RPM of 1200. The short fibres were refined to the maximum achievable specific refining energy (SRE) of 1059kWh/t with samples taken at 4 energy levels (283, 470, 826, 1059kWh/t). The refined short fibres were subsequently combined with the long fibres at a 44:56 short to long fibre mixture ratio. Additional combinations of refined short fibres to long fibres were also investigated ranging in composition from 0 to 100% MFC. For a comparison, the feed pulp was directly refined under almost the same LCR conditions as above, achieving a higher maximum SRE level of 1239kWh/t.

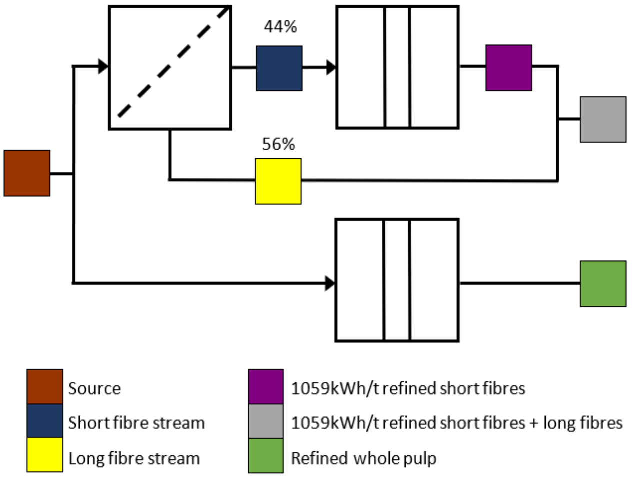


Figure 1. Experimental layout. The feed pulp was fractionated with only the short fibres highly refined and recombined with the unaltered long fibres. Separately the source pulp was also directly highly refined as a whole pulp.

PROJECT 2.3

Results

Figure 2a shows the decrease in bulk with SRE in the whole pulp (green line) compared to mixed pulps of long fibre fraction with varying portions of MFC (black and grey lines). The long fibre fraction (yellow square) has significantly higher bulk than the feed pulp indicating that the coarse, long fibres create a much more open and porous sheet structure. The short fibre fraction (blue square) has a lower bulk than the whole pulp. The higher bulk of the long fibre fraction over the refined whole pulp is maintained as the portion of added 1059kWh/t MFC in the combined pulp increases (grey line), up until the SRE reaches nearly 1000kWh/t. At the 1059kWh/t MFC addition of 44%, equal to the mass accept ratio of the fractionation process, the bulk is shown to be nearly 20% higher than the refined whole pulp at the same SRE. The long fibre fraction recombined with refined short fibre fraction of varying SRE in the same ratio as the fractionation process (black line) show increasing gains in bulk over the refined whole pulp with increasing SRE. The fractionation, short fibre refining and subsequent recombination at any ratio with the long fibres results in a higher bulk at the same SRE than conventional refining of the whole pulp.

Figure 2b shows that the long fibre fraction has significantly lower tensile strength than the unrefined whole pulp and the short fibre fraction has a significantly higher tensile strength. The tensile strength of the long fibre fraction combined with varying amounts of MFC is shown to be almost equal to the refining of whole pulp at SRE of approximately 500kWh/t or at the same ratio as the fractionation process (44% by mass of MFC). For the 56% long fibre and 44% refined short fibre combined pulps the tensile is nearly equal to, but less than the refined whole pulp. No combination of the fractionated and short fibre refined pulps exceeded the tensile strength of the refined whole pulp.

Figure 2c shows the fractionated, short fibre refined/MFC blended with the long fibre fraction can have significantly higher bulk at required tensile strength. The highest bulks are achieved for blends where the mass of MFC added is less than the mass of short fibre removed during fractionation. However, in a continuous process the remaining short fibre would have to be sold as a separate high value product. For blends where the short fibres are refined and recombined with the long fibres at the same ratio as the fractionation process there is some bulk increase at a given tensile, but the increase is less compared to the blends which have a smaller portion of short fibre MFC.

Figure 2d shows the decrease and subsequent increase of freeness with SRE in the whole pulp and in the blends of MFC with the long fibre fraction. The long fibre fraction has a significantly higher freeness while the short fibre fraction has a very low value. The high freeness of the long fibres elevate blends with the refined short fibre above refined whole pulp when the mass of MFC added is less than the mass of short fibres removed during fractionation. At the MFC addition of 44% to the long fibre fraction, the same ratio of the fractionation process, the blended pulps and the refined whole pulp show similar freeness values. At high SRE, the freeness values rapidly change from low to high, a result of the very small particle size passing through the mesh of the freeness tester. Higher values in freeness, in comparison to refined whole pulp, are only gained in blends which have a smaller portion of short fibre MFC.

Figure 2e shows the fractionated, refined short fibre/MFC blended with the long fibre fraction can have significantly higher bulk at required freeness values. The highest bulks are achieved for blends where the mass of MFC added is less than the mass of short fibre removed during fractionation. As described above this leads to an excess of short fibres. Blends at 44% MFC added to the long fibre fraction have some bulk increase compared to the refined whole pulp at a given freeness value, but the increase is less than the gains found in blends with a lower MFC content.

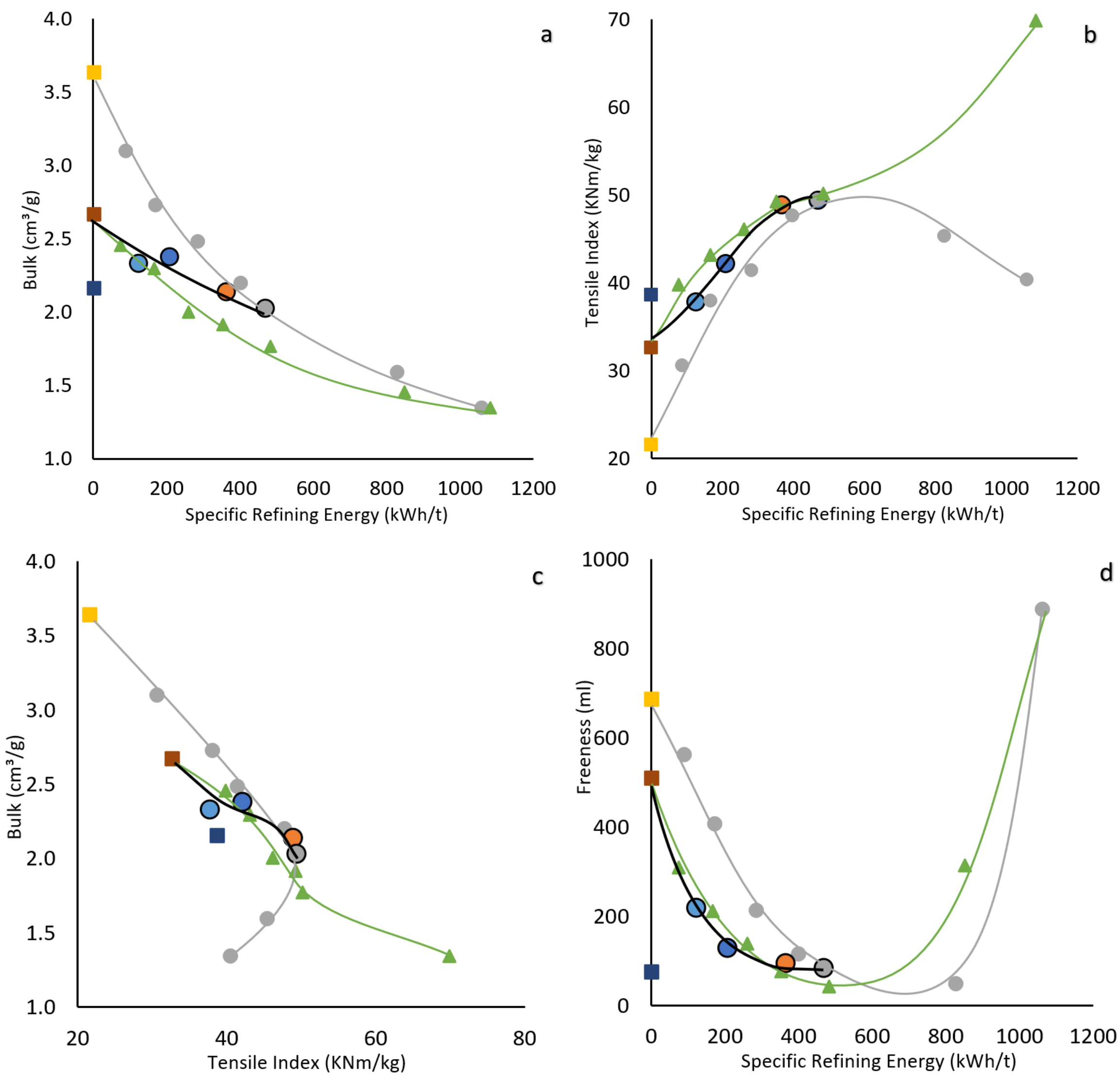
Conclusions

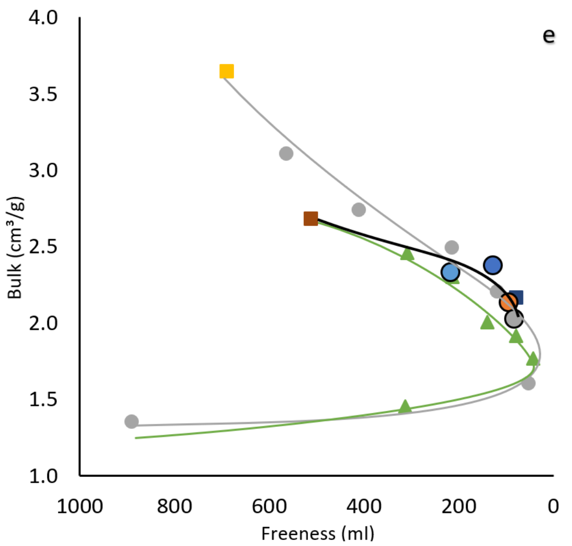
1. Fibre fractionation with small hole pressure screen can produce a long, coarse fibre that results in an open, porous, high-bulk sheet structure.

2. The addition of highly refined short fibre (MFC) can improve the strength of a high-bulk pulp resulting in a higher bulk, lower energy pulp.

3. Fractionation and short fibre refining can improve the bulk-tensile, bulk-freeness and bulk-SRE properties of mechanical pulp.

PROJECT 2.3





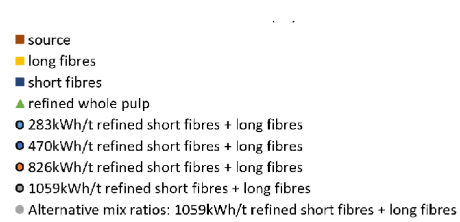


Figure 2. Comparison of mixed pulps to refined whole pulp. (a) bulk vs SRE (b) tensile index vs SRE (c) bulk vs tensile index (d) freeness vs SRE (e) bulk vs freeness.

PROJECT 2.3

Future research

Low energy MFC production using enzymatic pre-treatments and LC refining: We will determine the effect of enzyme type, concentration, and treatment time on the specific energy and quality of MFC produced from TMP and Kraft pulps as well as from mechanically fractionated fines by LC refining. MFC fibre quality as classified through image analysis and rheology will be determined as a function of specific energy and refiner intensity (Li et al., 2013) for a wide range of pre-treatment conditions. The pre-treatment conditions will be optimized for MFC quality and cost. Furthermore, the LC refining conditions will be optimized for the enzyme pre-treatments (Hu et al., 2018). LC refining experiments will be carried out using the 16-inch, single disc, Aikawa pilot refiner located at the PPC in UBC.

MFC fibre quality measurement: We will establish the relationship between fibre morphology (length distribution, aspect ratio, and specific surface) and MFC rheology for the goal of online instrumentation for process control. We will use 2D and 3D fibre image analysis to determine the key properties of MFC fibres through scanning electron microscopy (SEM), transmission electron microscopy (TEM), and the X-ray micro CT system available at UBC's Pulp and Paper Centre (PPC). We will correlate the suspensions' rheological properties (Shafiei-Sabet et al., 2016) to MFC quality as determined by image analysis and the impact on composite strength, bulk, and barrier properties. MFC rheology can potentially be measured on-line and in real-time, and as such may provide quality control of the production of MFC necessary for industry implementation.

Impact of MFC on fibre composite properties: The strength, bulk, and surface properties of composite sheets made with TMP and/or kraft and/or fines based MFC fibres and market TMP fibres will be determined and compared with conventionally LC refined sheets. This will provide an empirical understanding of the impact of MFC properties and content on the final composite properties. The detailed microstructure of the MFC-TMP fibre composites will be determined using X-ray microCT imaging, SEM, and TEM to develop a clear understanding of how MFC binds to the TMP fibres and enhances strength while preserving the bulk of the composite, and how MFC increases both the tensile index and tear strength, when there is normally a trade-off. This microstructure determination can form the basis for complex materials modeling of these composite materials.

MFC coating of TMP fibre sheets: MFC can be applied to the conventional TMP sheets to improve strength, surface, and barrier properties. The MFC coating will be applied through a number of processes including spray coating, bar coating, and several variations of roll coating (Beneventi et al., 2014; Lavoine et al., 2014). We will determine the impact of MFC coating of conventional TMP paper sheets on the strength, bulk, surface, and barrier properties of these sheets. We will develop an empirical model of the coating thickness and penetration as a function of MFC rheology and coater operation and design. A novel technology of using a micro-bubble foam to dewater and transport of the MFC fibres to the TMP fibre substrate will be developed and its effectiveness to enhance the surface properties will be evaluated.

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ELISA FERREIRA

3.1 - CREATING BULKY FIBRES

Background section

Fibre collapse occurs in papermaking during drying processes, in which the cell wall is affected by surface tension and external compressive forces (Mark et al., 2002). Collapse is characterized by a decrease in the lumen volume (Mark et al., 2002; Yamauchi, 2007) that alters fibre morphology to a flattened cross-section, thus reducing bulk. For the development of high bulk (low density) products, it is essential to understand and prevent collapsing mechanisms in fibres.

Since fibre collapse is controlled by surface forces, modification of fibre surface chemistry can be an effective route to create bulky fibres. The binding of amphiphilic and/or hydrophobic compounds is known to be effective to reduce the surface tension of cellulosics, with several approaches such as esterification, silanization, polymer grafting, etc. (Habibi, 2014; Hubbe et al., 2015). For pure and accessible cellulose, surface modification can be obtained through one-pot methods with minimum purification steps. One-pot protocols are shown to reduce the surface tension of cellulose nanocrystals and cellulose nanofibrils, based on the adsorption of surfactants via electrostatic interactions (Syverud et al., 2011), the irreversible adsorption of polymers (Hu et al., 2015), or the binding of polyphenols coupled with alkylamines (Hu et al., 2017)

This project aims to apply surface modification in thermomechanical pulp (TMP) fibres to prevent collapse by reducing the interfacial tension inside the lumen. TMP fibres have high lignin content (Mleziva & Wang, 2012) so that the amount of accessible cellulose must be enhanced prior to chemical treatments. Physical refining and NaOH mild treatments can contribute to accessible cellulose, as well as improve paper bulk (Choi et al., 2016). Besides the accessibility of cellulose, to ensure that the modification occurs inside the lumen, adsorption times must allow for penetration of the chemical compounds to reach the lumen, as previously described for the adsorption of polyallylamine (Gimåker & Wågberg, 2009).

Future research

Mechanical and chemical treatments will be systematically applied in TMP fibres. The effects will be evaluated with fibre and paper characterization and 3D microtomography imaging. TMP fibres will be modified through the following treatments:

Mechanical (physical) treatments: Refining will be applied to increase the accessible cellulose in fibres for chemical modification. Besides cellulose accessibility, mechanical processes are expected to affect other fibre parameters such as flexibility, length, and fibre-fibre interactions.

Chemical treatments: After physical modification, chemical treatments will be applied to bind hydrophobic compounds, surfactants, or polymers on fibres. All routes proposed are one-pot and water-based, allowing for three “mix and go” approaches:

(i) Hydrophobization reaction using plant polyphenols as a chemically-reactive primer, followed by the addition of amine-terminated hydrocarbons (Hu et al., 2017);
(ii) Adsorption-based modification of anionic (oxidized) fibres with cationic surfactant (cetyltrimethylammonium bromide) (Syverud et al., 2011);
(iii) Irreversible adsorption of methylcellulose (Hu et al., 2015).

PROJECT 3.1

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PROJECT 3.2

3.2 - ADVANCED CHARACTERIZATION – COMPUTED TOMOGRAPHY

Background section

This project will support most of the other projects in this consortium through development of advanced quantitative microscopy. Of key scientific interest is to develop novel imaging protocols to support the process-oriented studies in the other themes in this consortium. Recently, our group has developed innovative methods for characterizing the strength and structure of paper products using novel 3D X-ray tomographic microscopy (μCT) imaging and imaging-based modelling. The research at UBC has led to the development of novel automated μCT fibre segmentation algorithms in paper samples (see Figure 1). Fibre segmentation allows access to the properties at the fibre level such as the length, cross-section area and orientation as well as the properties at the network level including the coordination number of the fibre-fibre bonds. Although segmentation is a widely-studied topic, most work to date has focused on the application of semiautomatic techniques to separate only a few phases (less than 10) in the 3D images (Aronsson, 2002; Adams & Bischof, 1993; Axelsson, 2007; Svensson & Aronsson, 2003; Udupa & Samarasekera, 1996). The characterization of a fibrous network requires automated and robust segmentation of a large number of tortuous 3D fibres in large datasets. The most successful approach for segmenting papermaking fibres and other complex tubular structures is based on using the lumen as markers for fibre segmentation since they are separated from the background by means of thick fibre walls. Aronsson (2002) applied an ordered Region Growing algorithm to segment papermaking fibres from a 3D dataset obtained by SEM and microtomy, in which the lumens were grown from user-selected seed points. Axelsson (2007) presented a similar method for tracking of fibres in their direction of local orientation. Sevensson and Aronsson (2007) later extended their previous work to include the cracks in the fibre walls based on fuzzy connectedness. While these methods have proven useful in segmenting hollow fibres, they require the user to select a number of seed points for each fibre in order to initiate the segmentation. Thus, the number of fibres that can be segmented is limited. The numerical algorithm developed by our group is the first method that is able to segment a large fraction (currently 60–70%) of papermaking fibres within the dense network structure of paper in an automated manner.

The algorithm is able to capture the true surface topology robustly as well as the tortuous morphology of papermaking fibres despite the numerous complexities present in the data.

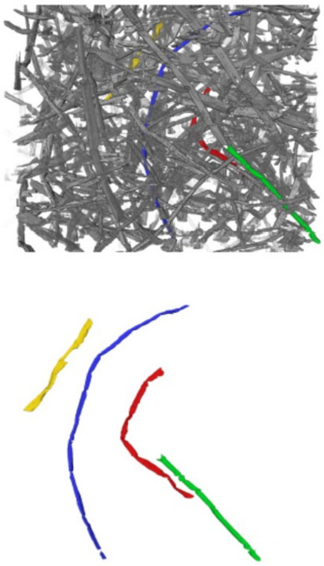


Figure 1. (a) Computed tomography of a standard handsheet (b) In the bottom panel, for 4 fibres have been segmented from the image and the properties analyzed.

We have recently used this imaging technique to characterize the "bulk potential" of a papermaking furnish. Here, we characterized the number of collapsed fibres in a paper sample and compared that to fibre wall thickness and lumen diameter of a wood chip. This is shown in Figure 2. Each point in this figure represents the fibre wall and diameter of individually imaged fibres. The red data represents the woodchip and the black data, a handsheet. The wood chip sample represents the maximum achievable bulk, as no fibres have collapsed. Ideally, if the bulk potential is retained, the data sampled in the paper sample would be superimposed onto the wood chip. Significantly, after processing, the bulk potential has changed and we find that thin walled fibres (Region 1, below the green line) are not evident and have collapsed to form Region 2. Seemingly, only fibres with a wall-thickness-to-diameter ratio of greater than about 0.1 remain open during processing. The immediate opportunity with this tool is to profile the mechanical pulping process to determine the loss in bulk.

PROJECT 3.2

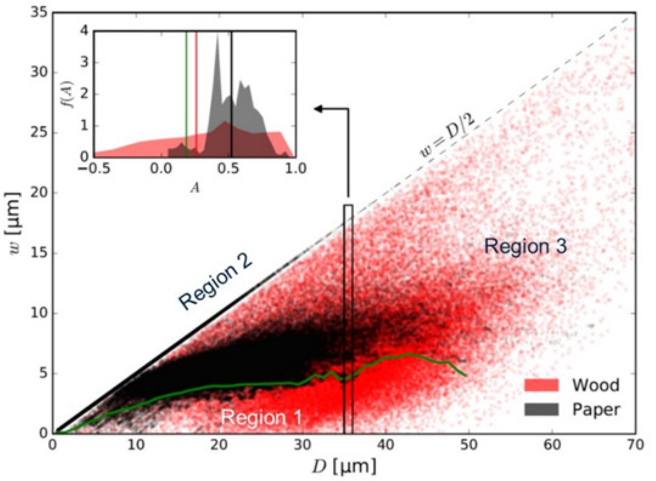
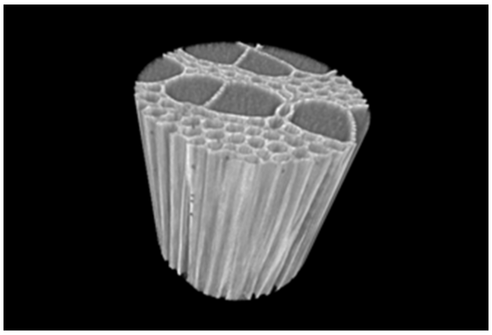


Figure 2. Using microscopic technics, we characterized fibre wall thickness w as a function of fibre diameter D for both a paper and wood chip.

Results

In the previous study, we developed an imaging protocol for a softwood wood chip. In the first phase of this work, we have updated the tomographic imaging protocol to examine an Aspen wood chip (adjacent figure). We were successful to image the chip at voxel size of 0.5 um over a field of view of about 1 mm3.



In the initial phase of this work, we are characterizing the change in morphology of the wood chip during sulphite treatments under conditions typically used in chemimechanical pulping (CMP) and chemithermomechanical pulping (CTMP). For the CMP type of treatment, Aspen chips were impregnated with 1 mol/L sodium sulphite solution at pH of 10 then cooked at 140^{o}C for 60 min. For CTMP type treatment, Aspen chips were impregnated in 0.4 mol/L sodium sulphite at pH of 12 then cooked at 90^{o}C for 30 min. Imaging of these treated chips is currently on going.

It is anticipated that under the milder CTMP type conditions most of the effects will be limited to softening of the lignin in the middle lamella as is seen with softwoods (Belgacem and Pizzi, 2016; Lönnberg, 1999). This is especially so as the more readily sulphonated guaiacyl lignin in aspen is concentrated in the middle lamella region. Under the more severe treatment conditions softening of the fibre wall is expected to occur. In hardwoods, such as aspen, carboxylic acid functional groups are also formed by hydrolysis of the esters of 4-O-methyl-D-glucoronic acid in the hemicellulose (Beatson et al. 1985, 1986). This is especially so at high pH. These grouping could also disrupt the fibre wall causing structural changes.

Hot water hydrolysis of Aspen chips was conducted to compare with sulphite pulping treatment at 140oC for 60 min. Due to the hemicellulose and lignin removal, cell wall distortion and shifting of the middle lamella were expected (Blumentritt et al., 2016). Under a similar hot water treatment condition of poplar, Ma et al. (2014) demonstrated by confocal Raman microscopy that lignin concentration throughout the cell wall decreases; the largest concentration decrease occurred in the secondary cell wall.

The next treatment of Aspen chips will be alkaline peroxide under conditions typical of alkaline peroxide chemithermomechanical pulping (APMP). For hardwoods, treatment with alkaline peroxide prior to refining has proven very beneficial for the production of bright strong chemithermomechancial pulps (Belgacem and Pizzi, 2016). As with the sulfite treatments, carboxylic acid groups are formed by alkaline hydrolysis of the esters in the hemicelluloses (Beatson et al. 1985, 1986). However, in this case, additional carboxylic acid groups will be formed by oxidation of lignin by the hydrogen peroxide. The generation of carboxylic acid groups can be expected to cause disruption of the fibre wall.

Further research is planned using softwood chips as the substrate and examining different parts of the mechanical pulping process.

PROJECT 3.2

Future research

We will continue the development of the image analysis algorithm and apply this to profile the changes in bulk across the mechanical pulping process. In parallel, we will continue our work on labelling and staining of fibres, after mechanical/chemical treatment given above, to enhance microtomography imaging. This technique will be used to track the motion of mechanical pulp fibres in a paper sheet during tensile testing and water absorption. Straightforward staining with osmium tetroxide will first be optimized. Alternative methods will examine fibre and paper coating with atomic layer deposition or chemical tagging. Recent literature has shown a number of metal nanoparticles that can easily be grown or deposited on cellulose and gold, silver, silica and iron nanoparticle synthesis on fibres will be tested for their ability to improve 3D X-ray imaging.

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ERMP PERSONNEL UPDATES

Farewells

Recently the ERMP group has said farewell to long serving staff members Meaghan Miller and Chitra Arcot as well as graduate student Hui Tian who has successfully completed her PhD program. We thank them for all their hard work and wish them the best in all their future endeavors.

Welcoming new arrivals

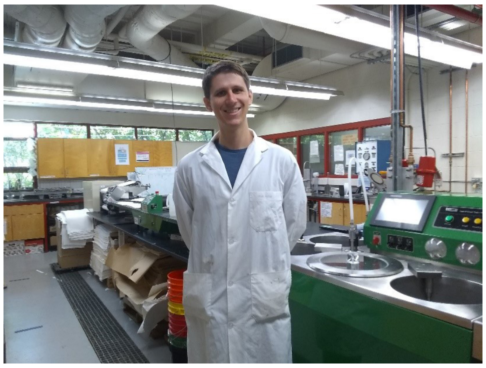
With the start of a new phase, we will be welcoming several new team members.

ERMP Program Manager, Daniela Vargas Figueroa

Daniela started as the new program manager for ERMP phase 3 in July 2020 after collaborating as a Researcher during phase 2. She holds a B.Eng. degree in Chemical Engineering from the University of Concepcion (Chile) and a MASc focused in Renewables Energies from the University of British Columbia. Her previous research included investigating the biochemical mechanisms of microbial enzyme action in industrial processing of plant biomass and its fermentation into bioproducts and optimizing dissolving wood pulp productions for improved chemical reactivity. As a member of the ERMP team, Daniela collaborated with Reanna on low consistency refining trials, and in sample analysis and testing of different cellulose microfibres as strength reinforcement for cellulose-based foams.

Daniela,
ERMP Program Manager.

To assist in the research activities Michael Bilek has joined the team as a laboratory technician in the papermaking lab. Michael is originally from a suburb of Chicago and earned a Bachelor's of Science in Biology from Eastern Illinois University in 2014 and a Master's in Forestry from the University of British Columbia in 2019. His research has focused on various aspects of sustainability, from pretreating biomass for gasification to selecting superior tree genotypes for fracking spill remediation. He was hired in the Pulp and Paper Centre in the fall of 2020 where his research focuses will include recycling of paper-plastic laminate waste and novel biofoam products.



Michael in the paper testing lab.

Graduate students

Claire Maulit

Claire Maulit, will be joining project 1.3 as a master student on November 1st. Claire graduated with a BASc from Notre Dame University and recently obtained a Diploma in Chemical and Environmental Sciences at BCIT where she gained theoretical and practical knowledge in pulping and papermaking.

Cameron Zheng

Cameron Zheng joined project 2.2 as a graduate student in October 2020. Mr. Zheng has a BSc Honours in Chemistry (University of Victoria, Canada). He has had numerous previous research experiences especially in the field of organometallics and organic synthesis. His bachelors thesis focused on the development of sustainable base metal catalysts for hydrophosphination. His current work in the Schafer group aims to apply hydroaminoalkylation towards the syntheses of biologically relevant compounds specifically focusing on the synthesis and modification of N-heterocycles. His expertise will extend toward the use of feedstock chemicals available from nature, specifically materials to be isolated and identified by post-doctoral fellow Pierre Betu Kasangana.

Cameron,
PhD student, project 2.2

ERMP PERSONNEL UPDATES

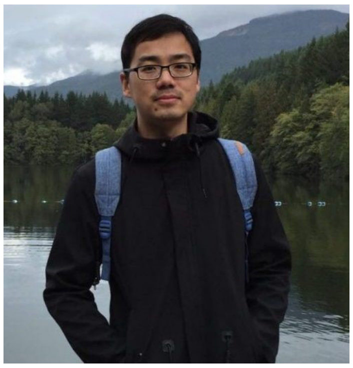
Postdoctoral fellows

Sudipta Kumar Mitra - project 1.3

Dr. Mitra obtained his PhD in Chemical and Biological Engineering from the University of British Columbia in 2020 under the supervision of Professors James Olson and Mark Martinez.

During his graduate studies, he examined tensile development during refining of mixtures of NBSK and hardwood pulp. He investigated the effect of separate refining and co-refining of mixtures of softwood and hardwood pulps in terms of tensile strength increase. He received his MSc degree in Pulp and Paper engineering from the Indian Institute of Technology Roorkee and received prestigious DAAD scholarship to carry out his master's project on ultrasound refining at Technische Universität Dresden, Germany.

Liyang Liu - project 2.1



Liyang Liu received his Ph.D. degree in Wood Science in 2020 from the University of British Columbia, working under the supervision of Prof. Scott Renneckar.

At present, he is a postdoctoral researcher at the University of British Columbia, working in the same lab. His main research interest focused on the greener chemical modification methods to convert the biomass (e.g., lignin) to platform resources for advanced materials, including foam, coatings, 3D printing inks, package materials, and electronic materials, as well as the advanced characterization methods of biopolymer such as nuclear magnetic resonance.

Pierre Betu Kasangana - project 2.2

Dr. Pierre Betu Kasangana joined the project as a post-doctoral fellow in October 2020. Dr. Kasangana has an MSc in Organic Chemistry (Université Pédagogique Nationale, R.D. Congo), MSc in Wood Science (Université Laval), and a Ph.D. in Wood Science (Université Laval).

His Ph.D. examined anti-diabetic bioactive molecules in the bark extracts of Myrianthus arboreus roots. He has been a PDF at Université Laval for the last two years; his recent work focused on the complex polysaccharides in sugar maple bark and black spruce as well as production of organosolv lignin from sugar maple bark. In addition, he has conducted work on the recovery of xylose from rice husks and wheat straw.

Elisa Ferreira - project 3.1

Elisa obtained a PhD in Chemistry in 2020 at the University of Campinas (Brazil) exploring lightweight materials prepared from cellulose and lignocellulosic fibres. Her past research focused on green approaches to prepare bio-based foams to be used in packaging, thermal insulation, and oil absorption. In the ERMP program, Elisa will investigate mechanical and chemical treatments to prevent collapse in fibres. She will systematically evaluate fibre processing and additives to control fibre intra-pore structure and to enhance bulk and absorbency.

Aurélien Sibellas - project 3.2

Aurelien Sibellas received his PhD in Mechanical Engineering in 2019 which was conducted in collaboration with MATEIS lab. (Lyon, France), MSsMAT lab. (Paris, France) and Michelin tires. He examined the mechanical behaviour of twisted textile yarns (reinforcements in tires) using both X-ray microcomputed tomography and simulation techniques. The same techniques were used in his previous postdoc to study rubber under compression. Due to delays created by Covid-19, Aurelien will begin his postdoctoral fellowship on Nov. 1st 2020.

LAB AND TRIAL UPDATES

In this transition time between Phase 2 and Phase 3 of the Energy Reduction in Mechanical Pulping program there has been a great deal of activity. The ERMP team has advanced our on-going projects while simultaneously expanding into the new research initiatives. Here is a quick snapshot of what has been transpiring in our facilities over the past year and a half.

New equipment

In 2019 the Pulp and Paper Centre upgraded our research capabilities with the acquisition of a Ziess X-ray Tomograph. This non-destructive testing device generates complete 3-dimensional imaging at the microscopic level, enabling our researchers to conduct in-depth analyses into the structural nature of their sample material. The tomograph has already proven to be highly successful and has motivated a future expansion of the Pulp and Paper Centre's other microscopic imaging equipment.

Zeiss Xradia 520 Versa Tomograph

Renovations

The Bioproducts Institute is implementing an extensive construction project at the Pulp and Paper Centre to update our facilities. Currently in the preparation phase, this project has already affected nearly the entire building and rejuvenated the workspaces by clearing out unused equipment and streamlining multistep pilot plant operations. The main demolition and reconstruction phase of the renovation project is soon to be underway with the work expecting to last for several months.

Trial updates

Since the last issue of this newsletter, pilot plant trials have continued when possible. Of major note were Matthias's rotary encoder refining runs, Bryan's pump efficiency tests and several operations centered around the production of micro-fibrillated cellulose. Between March and July of 2020 pandemic restrictions led to UBC closures and the suspension of these experiments. With a return to on-campus research and the commencement of ERMP's Phase 3 the pilot plant operations are planned to greatly expand over the coming months. All return to work activities at the Pulp and Paper Centre are being carefully designed, implemented and monitored to ensure the safety of all personnel in accordance with UBC coronavirus regulations.

Pilot plant refining trial with George Soong (top), Pranav (left) and Olivia (right). Image taken in 2019.

The Can-Mask Project

In response to the coronavirus pandemic and the subsequent shortage of personal protective equipment, several members of ERMP joined a large group of volunteers to contribute to a research program developing a prototype biodegradable face mask known as the Can-mask project. Utilizing ERMP's equipment, knowledge and experience our research staff have been helping to explore theories and experimenting with potential paper-based masks. Work on this endeavor is on-going with ERMP continuing to contribute to the project.

Experimental tests of the Can-mask fibre filters

PUBLICATIONS

We are pleased to announce the title of recent articles published in peer reviewed journals, and some of papers and presentations of our ERMP researchers.

Journal Articles

Villalba M., Olson J.A., and Martinez M., (2020): "The effects of Wood Chip Compression on Cellulose Hydrolysis". Submitted to Nordic Pulp and Paper Journal (under review).

Jahangir E.S., and. Olson J.A. (2020): "Low consistency refined ligno-cellulose microfibre: an MFC alternative for high bulk, tear and tensile mechanical pulp papers". Cellulose 27, 2803–2816

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Conference Papers and Presentations

Bohn B., "Practical Performance-Monitoring of Centrifugal Pumps", IEEE Sensors, Montreal, Oct 27-29, 2019. (Poster)

Bohn B., "Practical Performance-Monitoring of Centrifugal Pumps", Pacwest conference, Whistler, BC, June 6-7, 2019.

Villalba M., "Effect of Compression of Wood Chips on Cellulose Availability", Pacwest conference, Whistler, BC, June 6-7, 2019.

Aigner M., "Spatial Registration of Bar Force Profiles in Low Consistency Refining", Pacwest conference, Whistler, BC, June 6-7, 2019.

Jahangir E., and Olson J.A., "LC Refined Microfiber MFC Alternative for High Bulk, High Tear, and High Tensile Mechanical Pulps," 11th Fundamental Mechanical Pulp Seminar, Norrkoping, Sweden, April 2-4, 2019.

Theses

Hui Tian. "Stochastic Multi-objective Economic Model Predictive Control of Two-stage High Consistency Mechanical Pulping Processes", PhD Thesis, University of British Columbia, 2020.

Upcoming Event

Considering all new safety regulations related to Covid-19, the ERMP Steering Committee Meeting will be held online in November 6th, 2020.

We will notify soon all ERMP Steering Committee members with the meeting details in preparation for November.

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