On the usage of online fiber measurements for predicting bleached eucalyptus kraft pulp tensile index — an industrial case

R.B. DEMUNER, C.A. FARIA, A.A.R. DE ALMEIDA, AND P.N. PIGNATON

ABSTRACT: Cellulose pulp's physical-mechanical properties are determined by laboratory tests obtained from prepared handsheets. However, this procedure is time intensive and presents a lead time until the results are available, hindering its utilization for monitoring and decision-making in a pulp mill. In this context, developing real-time solutions for physical-mechanical properties prediction is fundamental.

This work applied a mathematical modeling approach to develop a soft sensor for tensile index monitoring. The mathematical model considers online morphology measurements obtained from the last bleaching stage outlet stream and important process variables for tensile index prediction. The results obtained are satisfactory compared to laboratory results, presenting a mean absolute percentual error of 2.5%, which agrees with the laboratory testing method's reproducibility.

Application: This paper presents the use of online morphology information to predict pulp physical-mechanical properties in real-time measurements.

Bleached eucalyptus kraft pulps (BEKP) are used in different paper product applications due to their unique physical-mechanical characteristics. For instance, in printing and writing applications, technical requirements for short fibers are related to improved printability, high bulk, high opacity, and high tensile index [1]. In tissue applications, the technical requirements are mainly related to softness and high bulk [2].

The fiber properties and other papermaking raw materials influence the properties of the end product. The wood supply and its processing in pulp mills are determinants [1,3]. The wood supply influences fiber morphology and chemical aspects. In this sense, the eucalyptus fiber morphology is peculiar. For instance, the fiber length is shorter than other species and narrowly distributed. The fiber coarseness is lower than other pulp fibers available on the market. Consequently, the fiber population is high. Additionally, the eucalyptus fiber's collapsibility is low due to a relatively high wall thickness [4]. Natural variation in wood supply is expected, which influences the morphological characteristics and, consequently, the pulp properties.

The relationship between fiber morphology and paper physical-mechanical properties has been a research topic for a long time. Page (1969) [5] developed a theoretical model for the paper tensile index. The fiber length and fiber width positively impacted tensile index, while coarseness affected it negatively. Additionally, the zero-span, the breaking stress of bonds, and the relative bounded area are other variables that influence the tensile index.

Page et al. (1985) [6] showed the effects of fiber deformation on pulp properties. For instance, increasing the fiber curliness decreases tensile index. This effect is related to reducing the actual fiber length used for bonding. One interesting point to highlight is that fibers are straight in wood. Thus, fiber deformation occurs during pulping processes. According to Page et al. [6], fiber deformation occurs due to mechanical and chemical effects' synergic actions. The mechanical effects may be unintentional and can happen, for instance, during high consistency pumping. Regarding the fiber deformation due to chemical effects, its occurrence has been reported during pulp bleaching in the caustic extraction stage [6].

Some research has been done using fiber morphology and other properties to predict pulp physical-mechanical properties. For instance, Horn (1978) [7] presented equations based on fiber morphology to predict hardwood fibers' properties. The main important variables considered were fiber length, length/cell wall thickness ratio, and fibril angle. In this study, eucalyptus fibers were not considered in the evaluation. Demuner et al. (1991) [8] presented a study on different species and hybrids of eucalyptus. It was concluded that wood basic density, fibers per gram, and pulp pentosans content were the main variables related to pulp physical-mechanical properties. Pulkkinen et al. (2008) [9] evaluated the correlation between fiber morphology and water retention value (WRV) and its impacts on physical-mechanical properties in eucalyptus fibers. Sundblad (2018) [10] applied previously developed models (e.g., Page (1969) [3] and Axelsson (2009) [11]) and estimated its parameters to fit models to laboratory data.

From the works mentioned earlier, fiber morphology is an important characteristic in describing pulp's physical-mechanical properties. Correlation and model development were already present in the literature. However, laboratory results are typically employed, and a limited number of samples are considered. Also, the performance of the models in an industrial application was not reported.

The traditional laboratory method of assessing pulp physical-mechanical properties is based on a beating device, such as the Norwegian Paper and Fiber Research Institution (PFI) mill device. Given different pre-established beating levels, the handsheets are formed and their physical-mechanical properties are evaluated at each level [12].

In pulp mills, these tests are done once or twice a week in the laboratory [13] and express the potential of pulps for papermaking, as the paper properties that might be produced on a paper machine are also dependent on paper production processes. Indeed, keeping the physical-mechanical properties stable in a pulp mill is important, consequently minimizing a source of variability for paper production.

However, due to the lead time of the conventional laboratory methodology, the results are available a few days after the pulp production time. Thus, the conventional method is not suitable for applications that aim to help decision-making during the production time.

It would be convenient and helpful to use the unbeaten fiber information available to predict pulp physical-mechanical properties, such that the potential of pulps for papermaking would be available. In this sense, Horn [7] showed that the unbeaten fiber morphology correlates well with physical-mechanical properties obtained from unbeaten and beaten fibers. Theoretically, it can be explained by hardwood pulp's physical-mechanical properties being primarily dependent on the characteristics involved in developing fiber-to-fiber bond potential, such as fiber flexibility.

Therefore, the present work offers a complete methodology to develop a soft sensor to predict the BEKP tensile index based on morphology and online measurements of process variables. This work also presents the results applied to an industrial operation of a large pulp mill placed in Brazil for over one year.

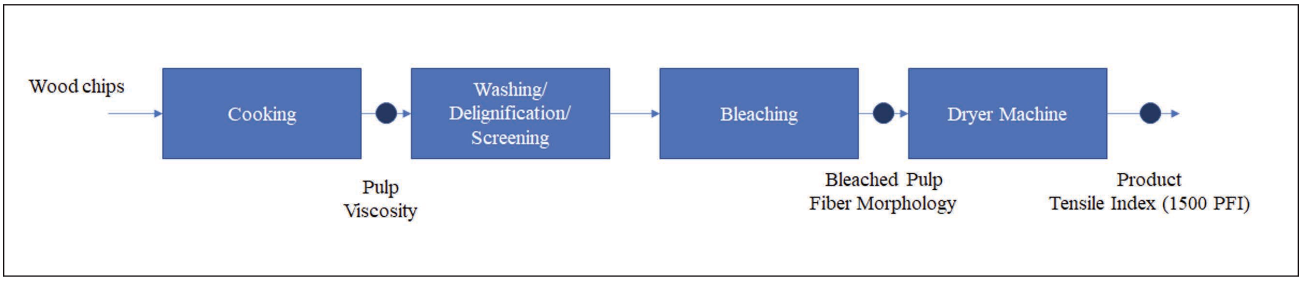
METHODOLOGY

Materials and methods

This section describes the methods applied to the tensile index soft sensor development. **Figure 1** presents the simplified process diagram and the positions of the measurements. After the dryer machine (dried pulp sheets), pulp samples were taken to assess their physical-mechanical properties by laboratory testing.

The pulp was disintegrated, beaten at 1500 revolutions in a PFI mill device, and the handsheets were formed and tested in the mill quality control laboratory. The methodologies considered are also presented in **Table I**. From the procedure described before, it was possible to obtain the tensile index at 1500 PFI revolutions, which was considered as the soft sensor output.

An online instrument for morphology measurement was installed at the bleaching plant outlet, which means that the morphology of never-dried pulp at a consistency of approximately 10% is sampled by the measuring device. New morphology information is available every 15 min. In terms of measurement technology, the fibers are analyzed through an optical method based on image analysis. A fiber suspension sample is collected from the bleaching plant outlet by an automatic sampler and sent to the measuring device. The samples are diluted in a dilution chamber using demineralized water in order to achieve very low consistency (0.004%–0.01% for hardwood pulps, according to ISO 16015-2 [2014] "Pulps — Determination of fibre length by automated optical analysis — Part 2: Unpolarized light method") suitable for fiber measurements. The sample then flows through the measurement cell, which measures only the amount of light received by the camera imaging cell. Due to scattering optics, the incident light is scattered from or absorbed by fibers in its path, and only the light that is not scattered will reach the camera. Therefore, the amount of light that does not reach the camera is equal to the particle area. This measuring concept allows the measurement of up to several million fibers, allowing repeatability of 1.5% for the average fiber length [14]. Based on image analysis, algorithms are applied to calculate morphology properties,



1. Simplified process diagram with measurements and sampling positions.

Analysis	Method
Morphology (fiber length & width)	ISO 16065-2 "Pulps — Determination of fibre length by automated optical analysis — Part 2: Unpolarized light method"
Laboratory wet disintegration	ISO 5263-1 "Pulps — Laboratory wet disintegration — Part 1: Disintegration of chemical pulps"
PFI beating	ISO 5264-2:2011 "Pulps — Laboratory beating — Part 2: PFI mill method"
Handsheet formation	ISO 5269-1:2005 "Pulps — Preparation of laboratory sheets for physical testing — Part 1: Conventional sheet-former method"
Tensile index test	ISO 1924-3:2005 "Paper and board — Determination of tensile properties — Part 3: Constant rate of elongation method (100 mm/min)"
Viscosity	ISO 5351:2010 "Pulps — Determination of limiting viscosity number in cupri-ethylenediamine (CED) solution"

I. Methods applied in laboratory and online equipment.

such as length, width, coarseness, fiber per gram, kinks, shape, mean kink angle, fines, and distributions thereof. The information generated by the equipment is then sent to the mill distributed control system (DCS) and process information management system (PIMS), where historical data can be consulted. Regarding the operating principle and specification details of morphology measurement equipment, the authors refer to ISO 16015-2 (2014), which establishes a standard for automated morphology measurements by optical sensors.

From the measurements obtained from the equipment, the following morphology information was considered for model development: mean fiber length (arithmetic), length weighted mean length, fiber width, fiber coarseness, fines, curl, mean kinks, and mean kinks angle.

In addition, the digester pulp viscosity was also considered a predictor variable. The viscosity is based on laboratory measurements. Table I presents the applied methodologies in this study.

Data acquisition and pretreatment

The tensile index results were measured daily, considering a composite sample from an interval of one hour of production, with a sampling time of 20 min. It means that three samples were collected during one hour on a given day, and this composite sample was tested.

To correlate the measured tensile index of the product to past process steps (e.g., bleaching and cooking), the retention time of each step was calculated based on the tank's volume and current level, pulp suspension consistency, and production rate of each step. The retention time value is considered as a lag, such that the morphology and viscosity data need to be time-shifted based on it.

Based on the sampling date and time of the product (pulp sheet), the corresponding morphology information at the bleaching plant outlet and pulp viscosity at the digester outlet are obtained based on the calculated retention time. This procedure can be interpreted as data synchronization, and it was applied to each product sampling instant available, which is always associated with a tensile index result.

The resulting dataset is a matrix of 363 lines (which represents the number of tensile index results) and nine columns, which represent the following variables: mean fiber length (arithmetic), length weighted mean length, fiber width, fiber coarseness, fines, curl, mean kinks, mean kinks angle, and pulp viscosity, which are considered as potential predictors for model development.

The resulting dataset represents a year of the mill operation and considers the pulp properties' natural variability, primarily related to the wood source. Also, model robustness is encouraged by sampling across the daily conditions, which may encompass different conditions and is not limited to a given operational condition of the mill.

Model development

The dataset previously obtained was randomly split, such that 70% of the data (254 points) is considered for model training (calibration) and the remaining 30% of the data (109 points) for model testing (validation). The data were scaled using a standardization scaler, such that the resulting data is centered on its average (zero mean) and scaled by its standard deviation. Mathematically, it can be written as follows:

$$x_{scaled,j} = \frac{x_j - \bar{x}_j}{\sigma_j} \quad (1)$$

where $x_{scaled,j}$ is the standardized j-th variable (j =1,…,m), m is the number of variables, x_j is the original j-th variable (without standardization), $\bar{x}_j$ is the j-th variable average value, and σ_j is the j-th variable standard deviation. For the sake of notation, this work considers the notation using the variable x for the predictor variables and y for the output (response) variable.

The data scaling step, here considered the standardization, is important due to the differences in the order of magnitude of the variables considered in the dataset. Thus, after standardization, the variables become dimensionless and in the same order of magnitude. Additionally, linear regression also allows for obtaining the standardized regression coefficients.

Multivariable linear regression was considered and implemented in Python code based on *Sklearn* library [15]. Thus, the model can be written as follows:

$$y_{scaled}^{m} = \sum_{j=1}^{m} \beta_j \, x_{scaled,j} \tag{2}$$

where y_{scaled}^{m} is the model output, here considered the standardized tensile index, and β_j is the regression model coefficient associated with the j-th variable.

In order to keep only independent variables in the dataset, a multicollinearity test was applied to remove variables that are strongly linearly related. In this test, the correlation matrix of the predictor variables is evaluated. If one pair of independent variables has more than 0.9 correlation, one of these variables is removed, preferably the one with a lower correlation to the output variable. The correlation evaluation was based on Pearson correlation coefficients.

Subsequentially, the model is trained using a backward feature elimination to select only statistical meaning variables based on a significance level of 0.05 for model parameters. The algorithm starts with all possible predictor variables and fits the model. The predictor variables' coefficients' significance values (p-value) are calculated, and the highest p-value is identified. If this value is greater than the predefined significance level, the corresponding predictor variable is removed from the training dataset. This procedure is repeated until all predictor variables' significance values are statistically significant.

The model coefficients are obtained by the maximum likelihood estimation problem [16], leading to a weighted least squares problem, such that the following objective function is minimized:

$$F_{obj} = \sum_{i=1}^{n_{train}} \frac{(y_i^m - y_i^{exp})^2}{\sigma_i^2} \tag{3}$$

where n_{test} is the test dataset size, y^m and y^{exp} are the tensile index predicted by the model and the laboratory data, and σ_i^2 is the i-th variance, which corresponds to the experimental error (variance) obtained in the i-th laboratory result.

In Eq. 3, it is important to notice that it corresponds to a generalization of the least-square problem. If the variance of each experiment is the same, the resulting objective function is equivalent to an ordinary least-square problem. In this work, the least-square objective function is assumed.

For model validation, the mean absolute percentual error (MAPE) was evaluated as model metrics, which is calculated as follows:

$$MAPE\ (\%) = \frac{100}{n_{test}} \sum_{i=1}^{n_{test}} \frac{|(y_i^m - y_i^{exp})|}{y_i^{exp}} \tag{4}$$

where $|\cdot|$ is the absolute value operator and n_{test} is the test dataset size. In Eq. 4, it is important to notice that the MAPE is calculated based on the tensile index value without scaling. Therefore, it represents the relative error itself, enabling the comparison of the model error to the laboratory reproducibility error.

Also, the model predictions were compared to the laboratory method based on a paired t-test considering the test dataset. The paired t-test is used to verify if the mean difference between pairs of measurements is zero. This test aims to validate if the method under development is equivalent to a reference method. In this case, this test is applied to compare the tensile index model predictions to the laboratory data. The authors refer to Montgomery [16] for details of the paired t-test.

In the paired comparison, the i-th paired difference is calculated as follows:

$$d_i = y_i^m - y_i^{exp} \tag{5}$$

The expected value of the difference is:

$$\mu_d = E(d_i) = \mu_m - \mu_{exp} \tag{6}$$

where μ_d is the expected value of the difference, E(·) is the expected value operator, and μ_m and μ_{exp} are the expected value of the model and laboratory results.

The null hypothesis is described as follows:

$$H_0 \colon \mu_d = 0 \tag{7}$$

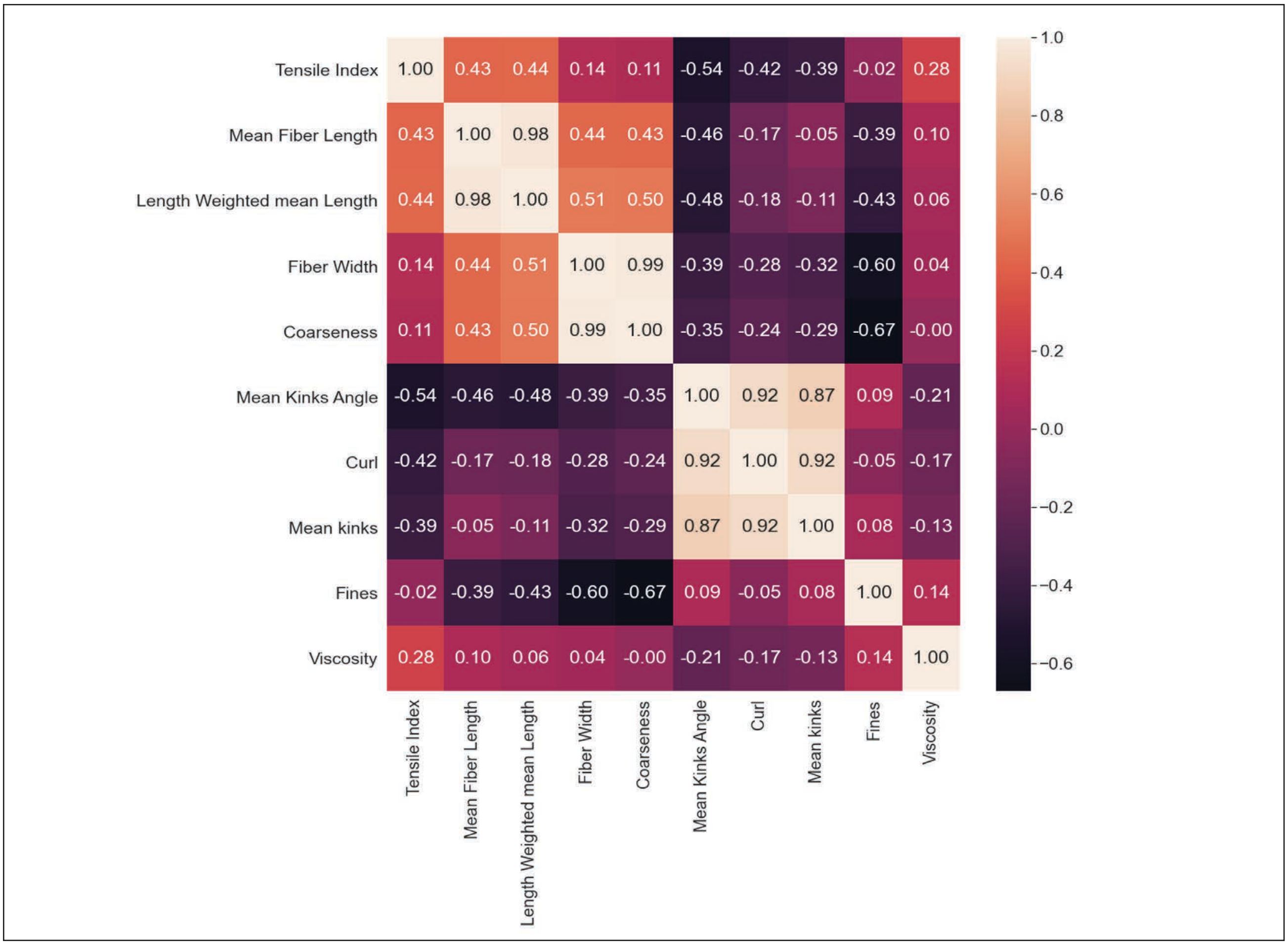
$$H_1 \colon \mu_d \neq 0 \tag{8}$$

The test statistic for this hypothesis is:

$$t_0 = \frac{\bar{d}}{s_d / \sqrt{n_{test}}} \tag{9}$$

where t_0 is the t statistics, $\bar{d}$ is the average of the differences, and s_d is the standard deviation of differences.

The average and standard deviation of differences are



2. Correlation analysis of fiber properties and tensile index. All variables are scaled following standardization.

calculated as follows:

$$\bar{d} = \frac{1}{n_{test}} \sum_{i=1}^{n_{test}} d_i \tag{10}$$

$$s_d = \sqrt{\frac{\sum_{i=1}^{n_{test}} (d_i - \bar{d})^2}{n_{test} - 1}} \tag{11}$$

H_0: $\mu_d = 0$ is rejected if $|t_0| > t_{critical}$, where $t_{critical}$ is the t-student value obtained for a significance level α and $n_{test} - 1$ degrees of freedom.

Additionally, the F-test is also applied to verify the regression model overall significance. The F-statistics is calculated as follows [17]:

$$F_{statistics} = \frac{MSR}{MSE} \tag{12}$$

where *MSR* and *MSE* are the mean sum of squares due to regression and the mean squared errors calculated as follows:

$$MSR = \frac{\sum_{i=1}^{n_{test}} (y_i^m - \overline{y_i^{exp}})^2}{p} \tag{13}$$

$$MSE = \frac{\sum_{i=1}^{n_{test}} (y_i^m - y_i^{exp})^2}{n_{test} - p - 1} \tag{14}$$

where p is the number of model coefficients, and $\overline{y_i^{exp}}$ is the average value of laboratory results.

In the F-test, if $F_{statistics} > F_{critical}$, the regression model presents overall significance. In this test, $F_{critical}$ is obtained from the F-distribution with a significance level of 0.05 and degrees of freedom equal to p and $n_{test} - p - 1$.

RESULTS AND DISCUSSION

Correlation analysis

The correlation between fiber properties and tensile index is presented in **Fig. 2**. It is possible to notice that the mean kink angle, curl, shape, and mean kinks have a negative impact on the tensile index. Indeed, these effects were already identified in the literature, and using the available

Variable	Coefficient	Standard Error	p-value
Mean kink angle	-0.3550	0.060	0.000
Length weighted fiber length	0.2435	0.061	0.000
Pulp viscosity	0.2474	0.056	0.000

II. Model coefficients, standard errors, and p-values. The coefficients are related to standardized variables.

online technology, it was also possible to see this effect. Additionally, the fiber length and viscosity correlate positively with tensile. The viscosity effect could be interpreted as a higher cooking selectivity, preserving carbohydrates and probably integrity of fibers. The effect of fiber length could be interpreted as a higher effective length for bonding. Also, the correlation between the tensile index and the variable's fines content, coarseness, and fiber width were low. Regarding the low correlation between fines and tensile index, as the morphology data is related to the unbeaten pulp, these fines are primary fines, which have a minor impact on the tensile index compared to secondary fines.

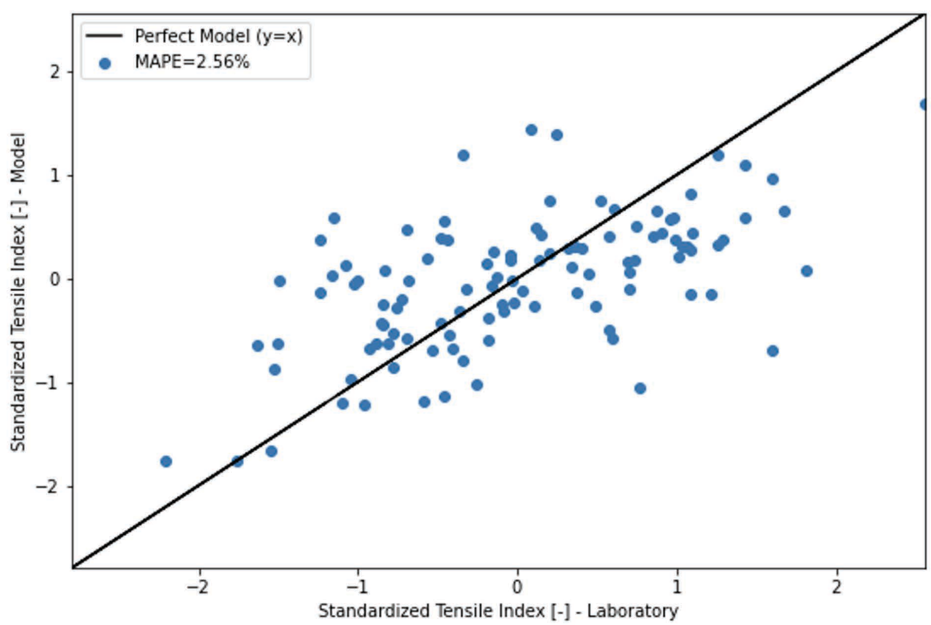
When analyzing the correlation among predictor variables, it can be noticed that the mean fiber length (arithmetic) and length weighted mean length are strongly correlated. It can be justified because these properties are based on the fiber length and are only calculated by different mathematical expressions. The same behavior can be noticed in fiber width and coarseness. It can be explained that coarseness depends on fiber weight, which depends on fiber width and cell wall thickness. Regarding the fiber shape variables, it can be noticed that there is a strong correlation among mean kinks angle, curl, and kinks, which is expected, as these variables describe fiber deformations.

Model training

After analyzing the correlation matrix, some variables were eliminated to avoid multicollinearity among predictor variables. The remaining variables in the matrix are the length weighted mean length, fiber width, mean kinks angle, fines, and pulp viscosity.

The multivariable linear model was fitted using the backward feature elimination procedure. The selected variables and their coefficients are presented in **Table II**.

From Table II, it can be noticed that only three variables were selected, which are: mean kinks angle, length weighted mean length, and pulp viscosity. Analyzing the coefficient signals shows that the mean kinks angle affects the tensile index negatively, while length weighted mean length and pulp viscosity affect the tensile index positively. This result agrees with the correlation matrix presented in Fig. 2. Also, as expected, due to the backward feature



3. Scatter plot of tensile index – model prediction and laboratory data in test dataset (time independently). MAPE is the mean absolute percentual error.

Mean of Differences	Standard Deviation of Differences	Standard Error of Differences	95% Confidence Interval of the Difference		t-statistics	p-value	Degrees of Freedom
			Lower	Upper			
-0.025	0.752	0.072	-0.144	0.094	0.34	0.72	108

III. Paired t-test applied for model predictions and laboratory method.

elimination, the p-value of each coefficient is below the significance value of 0.05 selected in this work.

Model validation

The scaled model prediction results against laboratory data are presented in **Fig. 3**. The predictions are calculated using the test (validation) dataset, which comprises 30% of the data gathered during a year of operation. The test dataset represents approximately three months of operation. It is important to highlight that Fig. 3 is time independent, showing an overall tendency of the validation period.

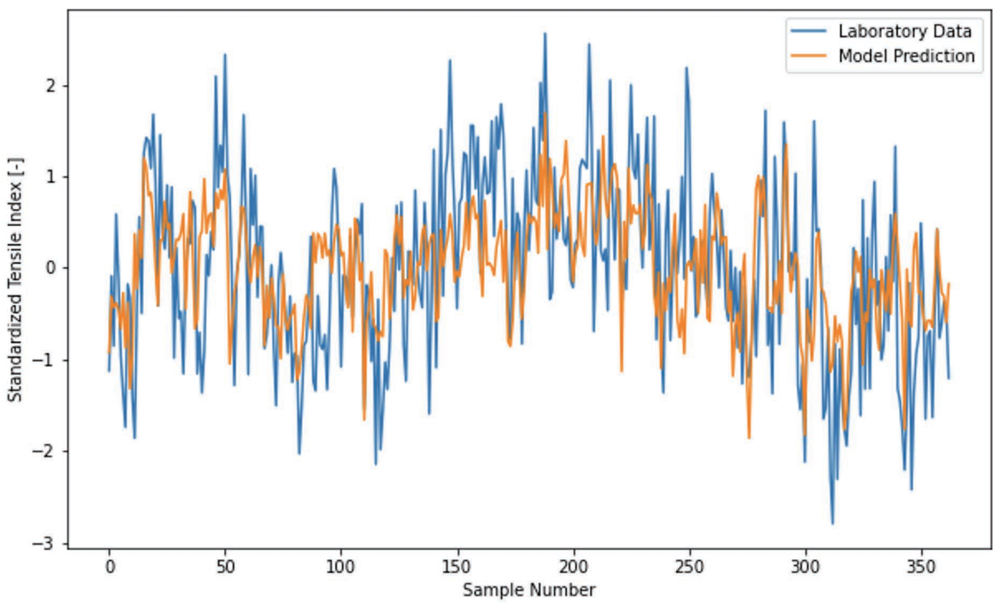
Analyzing Fig. 3 shows that the data is distributed along the perfect model line (y=x), which tends to fit the laboratory data. In the test dataset, a MAPE=2.56% was obtained. Here, it is worthwhile to mention that the laboratory method, ISO 1924-3 (2005) "Paper and board — Determination of tensile properties — Part 3: Constant rate of elongation method (100 mm/min)," indicates a reproducibility range of 2%–4%, depending on the paper grade [17]. Thus, the model developed showed an error comparable to the conventional method error.

Additionally, a paired t-test was carried out to compare model predictions and laboratory data. **Table III** presents the results obtained in the paired t-test.

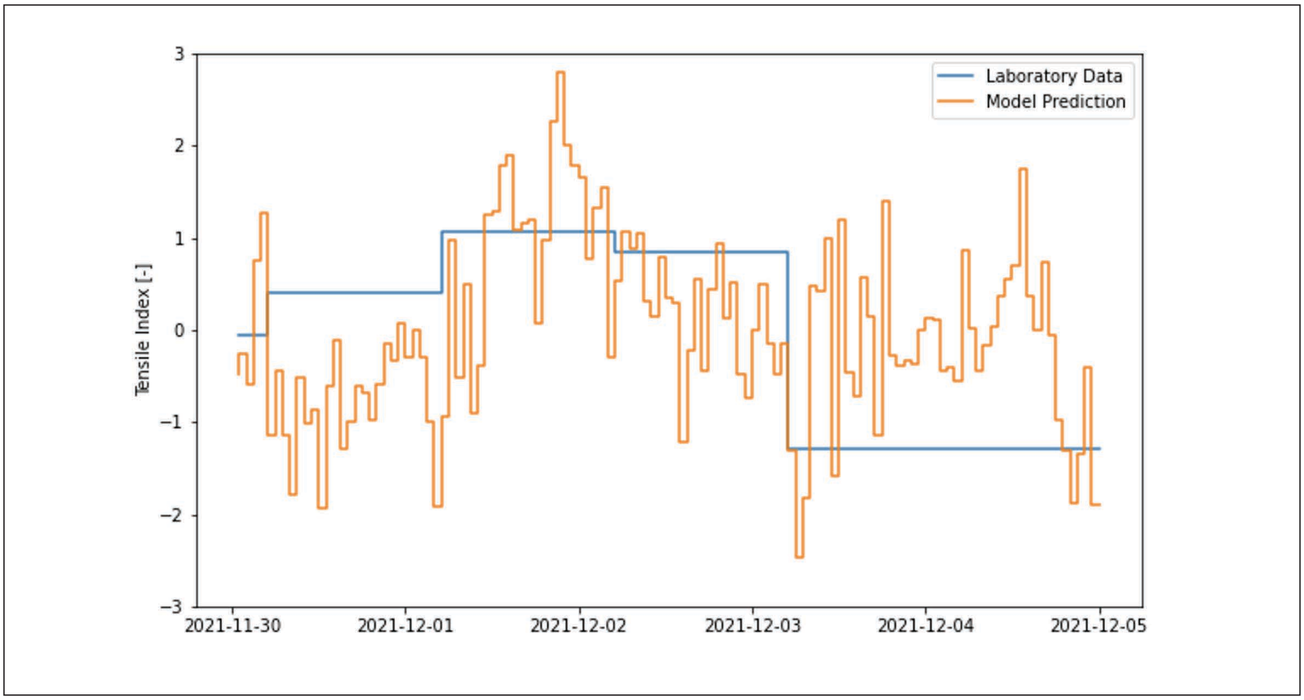
From Table III, it is possible to notice that a mean difference of -0.025 is obtained, with a confidence interval that contains zero. It means that, statistically, the mean of differences can be null, such that there is no difference between model and laboratory results averages. It can also be confirmed by the p-value that is higher than 0.05 (significance level). It means that the null hypothesis of identical averages cannot be rejected.

The regression model overall significance was assessed using an F-test. In the test data set, the calculated $F_{statistics}$ was 25.81, while the $F_{critical}$ = 3.24, considering a significance level of 0.05 and degrees of freedom of 3 (number of coefficients) and 105. Thus, as $F_{statistics}$ is greater than $F_{critical}$, it was concluded that the model has overall significance.

The time trend of scaled model prediction and laboratory results is presented in **Fig. 4**, which represents the predicted and laboratory tensile index results in the overall dataset (training and testing) and can be interpreted as almost one year of operation of the mill, as the tensile index



4. Time evolution of model predictions and laboratory data during a period of one year of operation.



5. Tensile index laboratory data and model predictions during a period of one week.

results are obtained on a daily basis. Analyzing Fig. 4 shows that the model predictions follow the laboratory data trends. There are some results peaks that the model cannot predict, which can be related to external variables that are not measured or considered by the model. However, the model can predict the trends of tensile, which is important for process understanding and its usage for decision-making.

One of the most important advantages of developing a soft sensor is data availability. As mentioned before, typically, physical-mechanical tests are carried out a few times during a week. By using a soft sensor, it is possible to achieve a higher frequency. **Figure 5** presents the scaled laboratory data and model predictions for one week.

By analyzing Fig. 5, it is possible to verify that model prediction at the time of the laboratory sampling agrees with laboratory results. It is also worth mentioning that the laboratory results are available at least six hours after the production time. In that sense, the soft sensor enables results on an hourly basis. Furthermore, using model predictions also enables verifying how the tensile index varies between the interval of laboratory results. This information also enables development of control actions and decision-making in a pulp mill aimed at tensile index variability reduction.

Discussion

Three variables were selected and identified as statistically significant regarding the fitted model: mean kink angle, length weighted mean length, and pulp viscosity. Although these variables are a consequence of wood source and process influences, this information could be further investigated to identify process variables strongly correlated to the model features. Thus, a causality map could be built aiming to leverage the tensile index.

The model predictor variables were identified through a data-driven strategy. The obtained relationships are also influenced by the conditions of mill processes and wood source. Direct extrapolation of this model to another mill needs to be carefully done since the output may be biased due to the differences in raw material and processes. However, it is important to mention that the identified variables agree with the theoretical foundation present in the literature and its effects. Thus, the variables are not limited to a specific mill, but corrections in model coefficients should be necessary when applied to another mill.

In this work, a considerable time range was considered for model development. A total of 363 tensile index results were considered, representing one year of mill operation, as the laboratory results are obtained daily. It means that natural variation of wood source and conditions of processes are considered.

Although validated by statistical tests, the model predictions presented an opportunity for accuracy improvement, especially in cases of tensile index peaks. It means that morphological variables do not fully explain the tensile index, and some additional information should be considered in the model. For instance, fiber chemical information, such as pentosans content, was found in the literature [8] as a variable of influence.

Despite the opportunity for accuracy improvement, the developed model can follow the tensile index trend, with a response time much faster than the laboratory results.

The main advantage of this model is data availability, which enables the mill operation to identify the tensile index fluctuations and take actions to minimize variability.

The following steps of this research intend to include fiber chemical properties into the model to improve its accuracy, as well as identification of key process variables aimed at minimizing tensile index variability. Moreover, variations in the wood mix due to the eucalyptus growth cycle need to be followed over time, and there is the need for a model recalibration due to these seasonal changes.

CONCLUSIONS

This work developed a soft sensor for predicting tensile index using online morphology measurements, such as mean kink angle, length weighted mean length, and the pulp viscosity. These variables agree with the theoretical foundation present in the literature and its effects. This work shows that the model mean average percentual error is equivalent to laboratory method reproducibility. Also, the averages of the soft sensor prediction results were equivalent to the laboratory results averages based on a paired t-test and overall model significance, which was assessed by an F-test. The results also showed an improvement in terms of data availability when using the soft sensor. **TJ**

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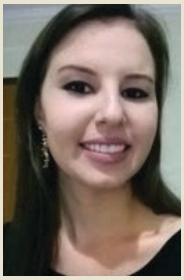
ABOUT THE AUTHORS

In this work, we developed a tensile index soft sensor based on online fiber measurements, which express the potential for papermaking of the pulp. The most challenging aspect of this research was getting accurate information from the process and treating it to generate information. We applied statistics tools for data treatment and mathematical modeling to develop the soft sensor.

From this research, we found the contributions of fiber morphology and process variables to strength. The soft sensor's main benefit is the availability of results compared to traditional laboratory measurements

The next steps for this research are related to incorporating fiber chemical properties into the model to improve its accuracy and identify key process variables aimed at minimizing tensile index variability.


Demuner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