# The influence of industrial process conditions on the quality properties of eucalyptus kraft pulp across the fiberline

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## Abstract

This work aimed at understanding the behavior of pulps along each step of the manufacturing process, obtained from industrial kraft Lo-Solids® pulping of eucalyptus chips, at a given kappa number (18.0 ± 0.5). Pulps were analyzed regarding morphological, chemical, optical and physical aspects. Similar xylose content was found across the production process. However, there was a fast refining development for brown and oxygen delignified pulps. In the first case, higher opacity and tensile index values were achieved with lower ºSR numbers, although higher amount of energy was consumed. After bleaching process, a reduction on those values was detected. There was no significant change for bulk property along the process. Considering fiber morphological parameters, a reduction of the average fiber length across the production process was verified, along with higher fines content and fiber deformations (curl and kink). No significant modification for total number of vessels was verified.

**Keywords:** kraft pulping; morphological; refining; tensile index; opacity; bulk

## Introduction

For the market pulp manufacturer, it's extremely important to understand the pulp behavior across de production process and its influence on the end product, in order to provide a good evaluation basis and prediction of the properties at the customers, meeting their requirements, regardless of the paper grade to be produced. For this reason, fiber general conditions in each production step is also very important, since it's exposed to very aggressive stages, from high pressure and temperature at the pulping process, through reaction with each bleaching agent, pumping and at storage tanks, until repetitive pressing operations at the dryer machine.

Some pulp quality properties are essential and are also considered key factors to distinguish different bleached eucalyptus kraft pulps, besides allowing process optimizations. According to Foelkel [1], the following characteristics are important: fiber population or number of fibers per grams of pulp (associated with fiber coarseness), individual fiber strength, fiber collapsibility, fiber bonding ability, fiber swelling and hydration, fiber deformations and pulp fines content.

During pulping and bleaching processes, fiber deformations like curl and kink occur, as well as modifications in the fiber structure, affecting pulp mechanical strength properties [2]. The authors complete that fibers with higher curl cause a reduction in tensile index, while kink affects mainly pulp wet strength. On the other hand, Foelkel [1] mentions that fiber deformations lead to significant improvements in paper porosity, bulk, absorption capacity and softness, characteristics which can be accelerated and artificially induced with proper device for pulp differentiation, when desired and advantageous, like for tissue papers.

Fibers with thicker cell wall in association with high density woods represent an advantage for water drainage during the papermaking process and lead to more porous sheets, with higher bulk and coarseness, less fiber population per pulp weight and, as a consequence, lower surface area available for interfiber bondings [3].

Among all the process steps for paper production, at the preliminary step of fiber preparation relies one of the most important points of study and evaluation in order to obtain desired properties: refining. Industrial refiner represents greater complexity than the laboratory one can simulate, with a wide number of parameters to be adjusted, like distance between disc surfaces, distance and inclination angle between disc blades, as well as its height and material selected.

Although pulp is considered a commodity product, paper manufactures are giving frequently greater value to product differentiation, looking for pulp desired characteristics required for each different final application. In conclusion, there are many pulp features to be assessed and kept under control, so that fiber engineering helps in technological development for different paper grades and their differentiation, from printing and writing, packaging and specialty to tissue papers.

In this context, the present study aims at understanding pulp behavior across the fiberline, applying kraft Lo-Solids® cooking and using eucalyptus as the wood source, in order to promote process optimizations and match paper requirements.

# Experimental

### Material

During the sampling period, LWARCEL CELULOSE pulp mill worked with *Eucalyptus grandis* wood chips, aged 7 years old and average 450 kg/m³ basic density. Lo-Solids® kraft pulping was used to achieve 18.0 ± 0.5 kappa number pulps. The cooking temperature was between 155 and 160 ºC, residual alkali was kept under control, with 4-6 g/l after impregnation zone and 8-10 g/l after the cooking zone. The other kraft pulping operating conditions at the pulp mill during the sampling period are described at Table 1. The ECF bleaching sequence was the following: O/O A/D PO D.

Six different samples were taken at different and subsequent production process stages, as follows: brown pulp after the first browstock DD Washer® (1st WAS), oxygen-delignified pulp after the last DD Washer® (LASWAS), first stage bleached and washed pulp (A/D), second stage bleached and washed pulp (PO), last stage bleached and washed pulp (D) and end product pulp sheets after the dryer machine (PRO).

Table 1: Industrial pulping conditions during the sampling period

| Pulping Parameters | Value |
|---|---|
| Effective Alkali, EA (%) | 16 |
| White Liquor Concentration (g/l) | 116 |
| Sulfidity (%) | 32 |
| H Factor | 670 |

### Methods

Tappi and ISO standard procedures were used for initial pulp evaluation regarding kappa number, viscosity and brightness (T236, ISO 302, T230, ISO 2470) as well as for refining at JOKRO mill, handsheets preparation and physical-mechanical testing (ISO 5264-3, ISO 5269, ISO 5267-1, ISO 1924-1, ISO 2471, ISO 534, ISO 536).

Fiber morphological measurements were obtained through a KAJAANI FS 300 device (fines particles were considered as bellow 0.2 mm) and for carbohydrates quantification, an ion chromatography was used, with hydrolysis procedure using T249 as a reference.

# Results and Discussion

### Initial Pulp Characterization

For the pulping kappa number established (18.0 ± 0.5), the results from kappa number, viscosity and brightness analyses from samples taken throughout the process, can be found at Table 2.

Table 2. Initial sample characteristics, regarding kappa number, viscosity and brightness analyses

| Sample | Kappa number | Viscosity (cP) | Brightness (% ISO) |
|---|---|---|---|
| 1st WAS | 17.9 | 50.2 | 33.7 |
| LASWAS | 10.0 | 34.2 | 54.7 |
| PO | 1.26 | 20.8 | 86.3 |
| D | 0.45 | 18.0 | 92.5 |
| PRO | 0.44 | 17.5 | 92.0 |

**Carbohydrates Content**

In order to provide support for interpretation and analysis from the pulp modifications occurred throughout the production process, the main carbohydrates were assessed. The two most important ones from eucalyptus (glucose and xylose) appear separated and the others are represented altogether (arabinose, galactose and mannose), for pulps at three different process stages: at the beginning of the brownstock washing (1st WAS), after the last bleaching stage (D) and the final product at the end of the drying machine (PRO).

Average carbohydrate analysis results, carried out twice, as well as statistical treatment employing F Test, followed by Tukey Test in the case of multiple comparisons between traits, at 5 % of significance adopted, are shown in Table 3.

Data dispersion was evaluated through the coefficient of variation (CV) from each of the experiments, as also shown in Table 3.

Table 3: Carbohydrates analyses results* throughout the production process, together with statistical treatment applied

| **Sample** | **Kappa Number *18.0 ± 0.5*** | | |
|---|---|---|---|
| | **Glucose** | **Xylose** | **Other Sugars** |
| 1st WAS | 83.53 | 12.34 a | 0.30 a |
| D | 84.49 b | 13.67 ab | 0.28 ab |
| PRO | 84.75 b | 13.13 ab | 0.57 ab |
| CV (%) | 0.12 | 3.49 | 35.95 |

*Mean values followed by the same letter at columns don't differ from each other, according to Tukey statistical test (α=0.05)

Table 3 shows there is an increase in glucose content from brown pulp (1st WAS) to bleached pulp (D), keeping then unchanged until the end product. This behavior can be explained by the fact that pulp, when brown, is comprised with greater amounts of other components, such as lignin, which is removed and modified along the process, notably at the bleaching plant, increasing carbohydrates share in pulp.

With regards to xylose and other sugars analyzed, there is no significant change throughout the process for the cooking kappa number studied, at 5 % significance level. This fact can be due to good carbohydrates preservation at modified pulping processes, like Lo-Solids® used in this study.

**Morphological Properties**

For a better understanding of fiber behavior throughout the process, its main anatomical dimensions were evaluated using KAJAANI FS300 fiber analyzer: length, width, fiber population, coarseness, fines content, vessels content and fiber deformations, through curl and kink properties.

Average analyses results, carried out twice, as well as statistical treatment applying F test, followed by Tukey Test in the case of multiple comparisons between traits, at 5 % of significance adopted, are shown in Table 4.

It can be inferred from Table 4 that, when considering mean fiber length, there is a reduction of this property after going across the fiberline, however showing an increase after the drying machine. This behavior can be explained by the fact there is a loss of small fibers and fines through the white water drained from the wet section formation zone, remaining at the pulp sheet formed and thus, at the end product, fibers with higher average length.

Table 4: Comparative morphological analyses results* along the production process, from brown pulp until the end product, for pulping kappa number 18.0 ± 0.5

| **Process Stage** | **FL** mm** | **FW** µm** | **COA** mg/100m** | **Nr Fibers/ Mg** | **Fines %** | **Curl %** | **Kink 1/m** | **Vessels (1/g)** |
|---|---|---|---|---|---|---|---|---|
| 1st WAS | 0.880 | 15.57a | 6.55abcd | 19704.5 | 3.57 | 9.9 | 949.6 | 114620abcd |
| LASWAS | 0.823 | 15.36a | 6.80abcd | 21115.3 b | 8.19 | 13.45 b | 1455.5 | 101390abcd |
| A/D | 0.790 | 14.93 c | 6.75abcd | 22211.4 bc | 6.78 c | 17.85 c | 2351.9 | 121185abcd |
| D | 0.763 | 14.80 c | 6.80abcd | 22699.5 cd | 7.08 cd | 19.20 c | 2639.8 | 113680abcd |
| PRO | 0.800 | 14.17 | 6.40abcd | 23254.0 cd | 6.79 cd | 14.67 b | 1631.3 | 119180abcd |
| **CV (%)** | **0.28** | **0.69** | **1.96** | **1.34** | **2.09** | **2.74** | **2.37** | **7.36** |

*Means followed by the same letter at columns don't differ from each other, according to Tukey statistical test (α=0.05). **Where: FL= Fiber Length; FW = Fiber Width; COA = Coarseness

Fiber width kept without significant changes along the brownstock washing (1st WAS to LASWAS), suffering a decrease after going through the last bleaching stage and another reduction after the end product, reaching 14.2 µm.

Coarseness property did not show significant changes throughout production process.

Regarding fiber population, from brown pulp to pulp after oxygen delignification stage, there was an increase in the number of fibers per grams of pulp and since the first bleaching stage until the end product, there was no significant change.

Fines content showed the lowest value for the 1st WAS pulp compared to all the following process stages. There was, however, a reduction after the A/D bleaching stage, remaining then without statistical difference until the end product.

According to Frinhani and Oliveira [4], it's well known that refining operation influences drainage and paper drying, a fact that also interferes with additives retention and their interactions with pulp fiber functional groups. The authors also mention that more intensive refining levels reduce fiber population per pulp weight, increasing as a consequence fines content, which leads to an increase in the surface area and fiber interactions.

Fiber deformations curl and kink increased from brown to oxygen-delignified pulp, the same observed from the former stage to A/D bleached pulp. Both deformation properties studied showed reductions from D bleached pulp to the final pulp sheets. This behavior can be explained by the fiber alignment that happens when the pulp suspension jet, with five times lower consistency than the storage tower, is directed to the wire from the wet section formation zone of the dryer machine, causing deformations reduction.

With regards to pulp vessels content, there was no significant change throughout the process, with average value of 113,500 vessels/g.

The importance of vessel content for the papermaking is related to the effect that may occur during the paper printing process, known as vessel picking. This term refers to the phenomenon where some vessel elements present on the paper surface tend to be removed through the adhesion effect by the printing ink, causing as a consequence some failed spots on the printed surface [5]. The authors mention that vessel elements may have different shapes that vary with wood species, the most prejudicial ones during the offset printing process being the widest and shortest, regardless if coated or uncoated papers are considered.

**Refining**

In the attempt to simulate pulp performance at the papermaking process and to enable to identify its properties along the production line, JOKRO mill experimental refining analysis was carried out, followed by optical and physical-mechanical handsheets testing. Brown, oxygen-delignified, D bleached and pulp sheets were analyzed in this case.

Opacity and tensile index versus ºSR drainage resistance results are shown in Figure 1. These two properties were chosen to be graphically represented given their great importance to Printing and Writing (P&W) papermaking. Opacity is important because it's related to the need for the text printed in one side of the paper to do not appear on the other side, a fact that would disturb reading and printing quality. Paper strength, represented in this case by tensile index, is required to deal with sheet tension both at papermaking and printing machine, turning into possible good runnability conditions.

For each of the graphs showed below (Figures 1 and 2), all the curves were statistically different by the identity models method [6], for 5 % probability assumed, which means they cannot be represented by one single regression equation. In other words, these properties studied vary along the production process stages.

There was a fast refining development for both alkaline pulps, brown and oxygen-delignified, reaching in the first case higher tensile values and opacity within lower ºSR numbers, although with higher refining energy demand, associated with higher refining time (Figure 2).

The above mentioned superior refining performance for both unbleached pulp samples when compared to all the following stages can be explained by the fact of working with alkaline conditions until the end of brownstock washing, which leads to fiber swelling and as a consequence better fiber bonding and pulp strength properties development. Moreover, higher opacity values are explained by the paperweb entrained structure.

As the pulp goes to the bleaching plant, where it suffers consecutive pH shocks and reactions with chemical agents, it ends up with lower tensile index and opacity.

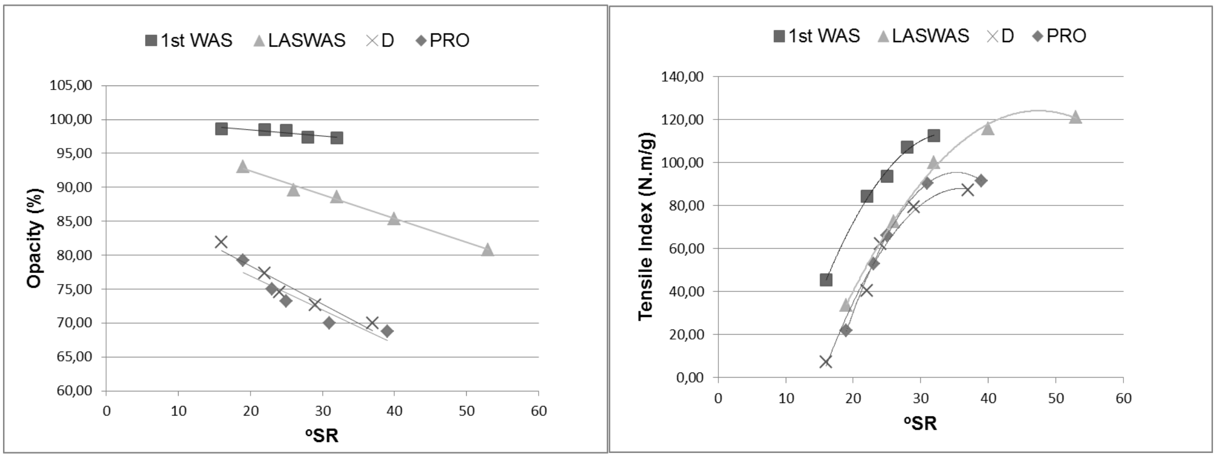

Figure 1: Opacity and tensile index development with refining, versus ºSR, at four different fiberline stages operating with target pulping kappa 18

In the end, when reaching the dryer machine, an increase in the tensile index was observed, simultaneously with a reduction in fiber deformations curl and kink. For higher ºSR values, there was a reduction in opacity, a fact that could be explained by the fiber deformations decrease, which leads to higher light transmission through the paper sheet, because of a greater number of empty spaces. The explanation for the strength improvement at this stage could be due to a previous acid last stage of bleaching, achieving low pH values (3.5-4.0), a condition that makes it more difficult to refine the pulp, comparative to still acid but higher pHs at the dryer machine, caused by white water drainage, removal and some change by fresher and cleaner water.

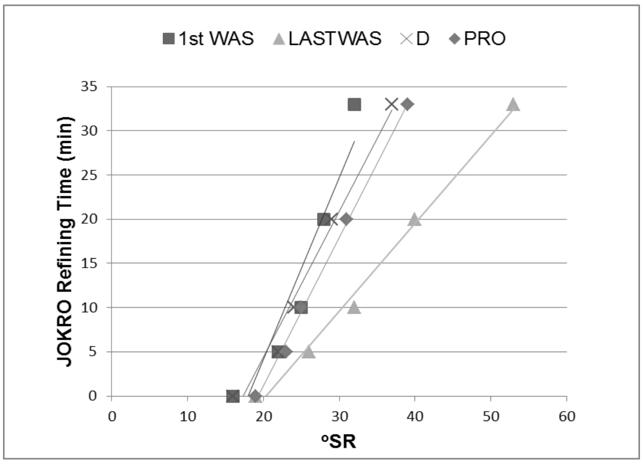

Figure 2: Refining energy consumption from pulp samples throughout the process, expressed as the refining time as a function of drainage resistance ºSR

Given the importance of this property for the production of tissue papers, bulk was also selected for further comparisons on this study, as it is usually associated with paper softness, extremely desired for the previously mentioned papergrade.

The identity models statistical treatment was once again applied for the development of bulk with refining, as a function of ºSR (Figure 3), which in this case indicated no significant difference between equations from each curve representing a different process stage, at 5 % probability. That means all the curves in this trait can be represented by one single regression equation.

In conclusion, bulk property did not show significant variation along the four production process stages assessed: 1$^{st}$ WAS, LASWAS, D and PRO. This result is very interesting for tissue papers.

However, it’s important to mention that the same bulk values along the process for each ºSR obtained, were achieved with different refining energy consumption (Figure 2), the lowest for the LASWAS pulp. This piece of information is relevant for papermakers, since electricity has a major impact in total production cost.

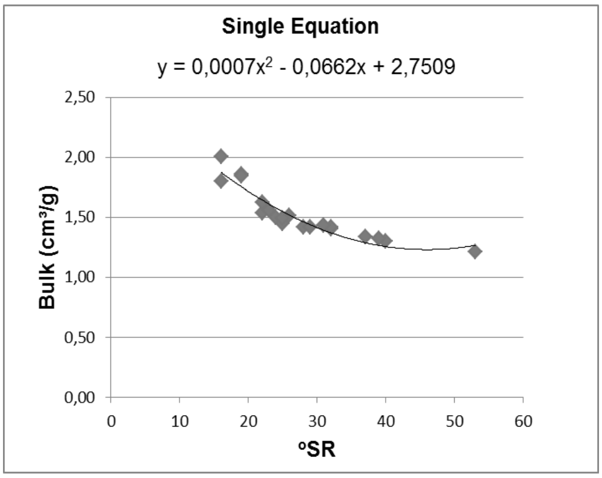


Figure 3: Bulk behavior as a function of ºSR, with all the curves statistically identical for the four process stages studied (1st WAS, LASWAS, D and PRO)

In order to help interpretation and understanding of all the properties studied along the production process simultaneously, Figures 4 and 5 were built, containing the main chemical, morphological, optical and physical-mechanical properties at a given 30 ºSR, for pulping kappa number target 18, considered as standard for this process studied.

Four process stages comparisons are represented, always assuming one stage compared with the previous one, except from the first case (oxygen-delignified pulp), when the reference as a previous stage was the 1st WAS pulp. When a value is equal to zero, that means there is no statistical difference between the compared stages.

Only xylose content (Figure 4) was not analyzed in all the four process points considered, with results only for D and PRO pulps

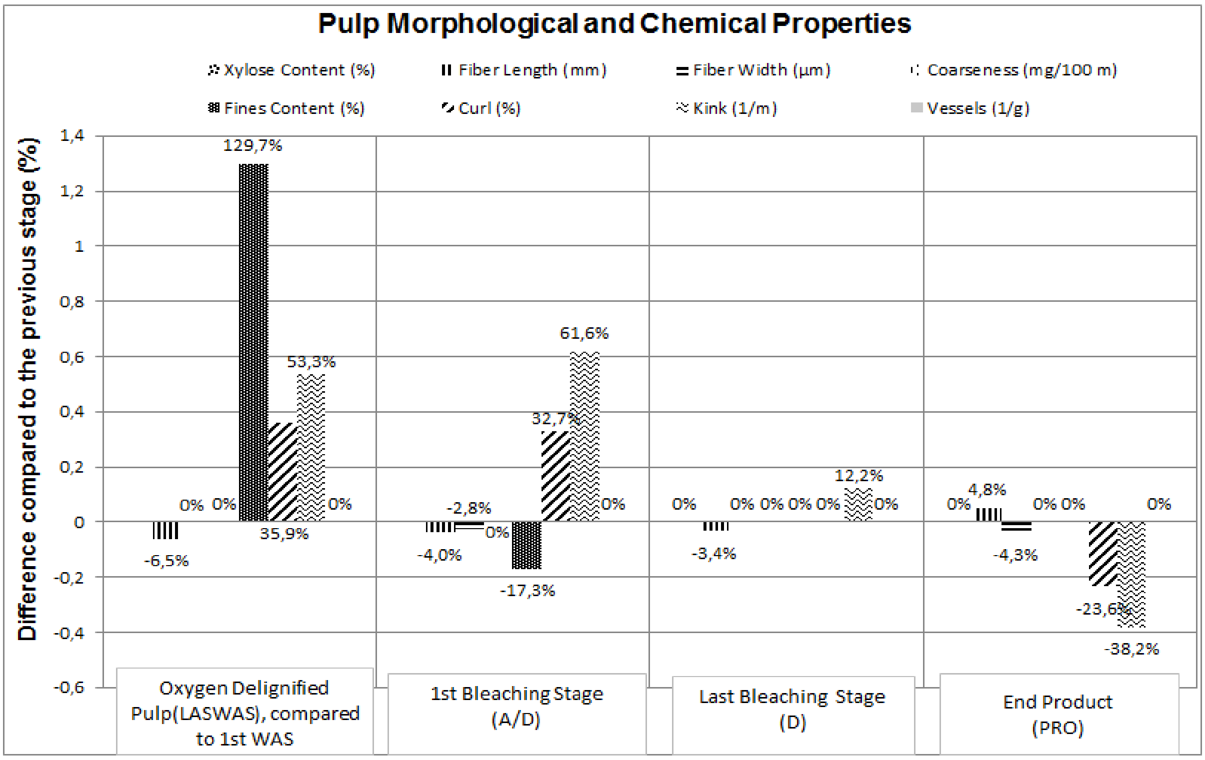


Figure 4: Comparative variation between process stages, regarding the main chemical and morphological pulp properties, using as a reference the previous stage

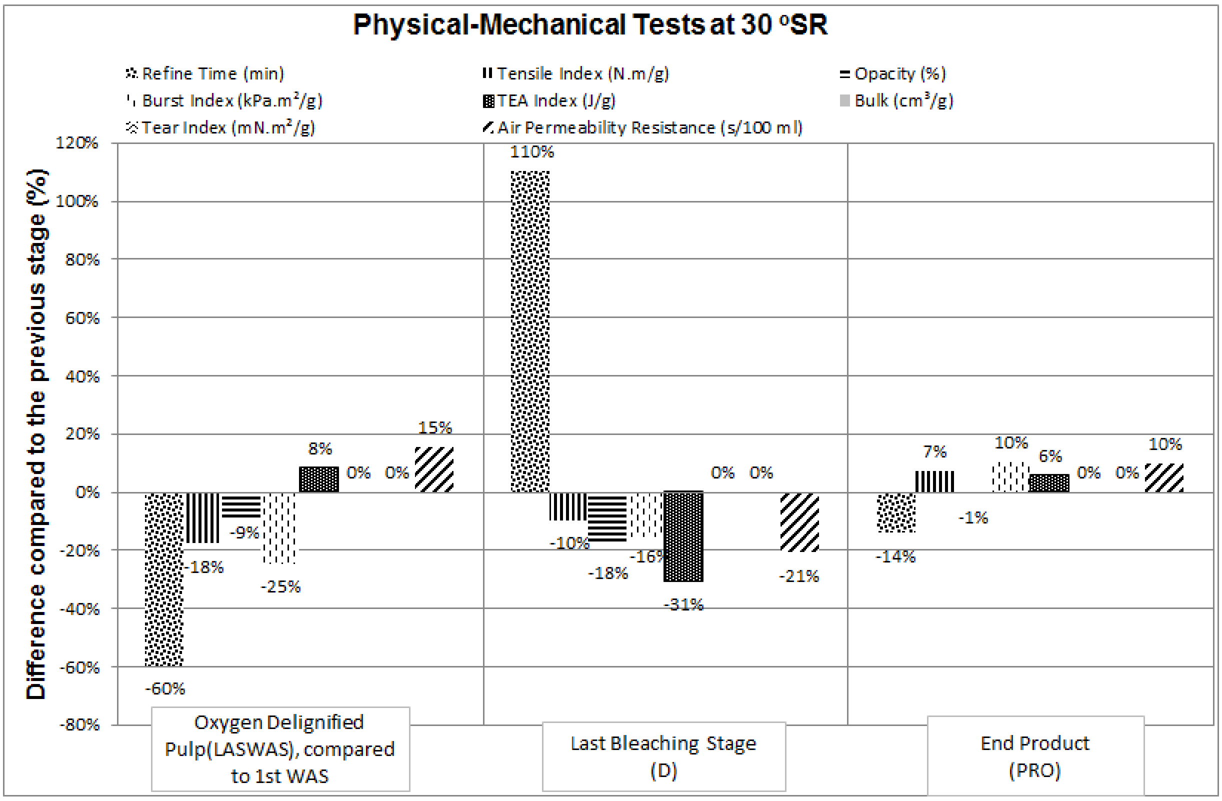


Figure 5: Comparative variation between process stages, regarding physical-mechanical and optical pulp properties refined at 30 ºSR, using as a reference the previous stage

Initially, Figures 4 and 5 show that the properties which had no significant variation in any of the production process stages considered were xylose content, coarseness and pulp vessels content, besides bulk at 30 ºSR.

A reduction from 1$^{st}$ WAS to LASWAS pulps can be noticed in fiber length (6.5%), strength property tensile index (18%) and opacity, around 10%. However, there was a notable decrease of 60% in refine time to achieve 30 ºSR, indicating an ease to refine pulp. A substantial increase of more than 100% in fines content and around 36-53% in fiber deformations curl and kink explain the strength loss.

Fiber deformations curl and kink continued to increase after leaving the first bleaching stage. At the same process point, a reduction of 4% in mean fiber length occurred, followed by strength and opacity losses from D pulp, with impressive increase of 110% in refine time to achieve 30ºSR. The reason for this is most likely the acid conditions from the last bleaching stage, that pulp has to face for two hours, turning refining more difficult. Kink continued to go up after the D stage (12.2%), as well as mean fiber length continued to go down (3.4%).

When comparing PRO pulp with previous D stage, curl and kink deformations decrease by 24 and 38 % respectively, indicating a consequence of fiber alignment at the wet section formation zone of the dryer machine. Mean fiber length increased 4.8% at the same process stage, which could be explained by the effect of small fibers loss at the white water drained from the formation zone, remaining at the pulp sheet and as a consequence at the end product, fibers with superior average fiber length. Meanwhile, there was a 14% reduction in refine time to achieve 30ºSR, with improvement in tensile index property.

Pulp sheet strength, expressed at this study mainly through tensile index, suffers consecutive reductions after facing aggressive process conditions like temperature, pressure and chemicals reaction at the stages of pulping, specially oxygen delignification (18%), besides bleach plant (10%). On the other hand, there is a small recovery after leaving the drying machine (7%).

## Conclusions

With the results of this study in hands, it can be concluded that chemical, morphological, optical and physical eucalyptus pulp properties presented significant and important modifications along the production process for the kappa number considered (18.0 ± 0.5), with special attention to oxygen delignification stage, detailed as follows:

1. There is no significant difference in the xylose and vessels content of the pulp throughout the process, as well as for bulk.

2. Regarding average fiber length, a reduction of this property occurred along the fiberline, presenting however an increase after the dryer machine, due to fiber alignment at the wet section formation zone, confirmed by the decrease of fiber deformations curl and kink.

3. Fines content showed an expressive increase from the post cooking brown pulp to the following stage, oxygen delignification (130%). The same behavior was observed for fiber deformations curl and kink, confirming aggressive conditions at the oxygen delignification stage that caused also decrease of the strength properties at 30ºSR.

4. Refining performance showed a rapid development for alkaline brown and oxygen-delignified pulps, achieving in the first case higher tensile index and opacity values with lower ºSR numbers, however with superior energy consumption. After pulp bleaching, there was a reduction in those values. The greatest tensile index reduction at 30ºSR occurred after oxygen delignification stage (18%), together with burst index (25%).

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