Impregnation of eucalyptus and pine wood in alkaline pulping processes. Effects of steaming and pressurized impregnation

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Chip Impregnation for pulping

A proper

- Penetration of liquids
- Diffusion of chemicals

Both are necessary for optimal efficiency of:

- Chemical pulping
- Chemimechanical pulping
Uniformity

The uniformity of the impregnation determines the uniformity of the chemical treatment in the first stage of pulping. Is essential for the homogeneity of the pulp obtained.
Distribution of kappa number of a kraft softwood pulp

Fluorescence on kraft fibers

Yue Liu, Tappi J. 82(9), 1999

3. AO stained kraft fibers with kappa nos. 14, 32, and 82 exhibiting green, yellow-orange, and red-orange colors, respectively
The Malkov work shows:

- A fairly broad distribution of kappa of the softwood kraft pulp.
- Distribution can be notably reduced when a proper impregnation is applied.
Benefits of a proper wood impregnation in a kraft pulping

In general it is shown in the literature that it can lead to:

- Increase in pulping yield
- Reduction in cooking time
- Reduction in bleaching chemical
The alkaline impregnation phenomenon is complex. **It implies:**

- Air removing
- Penetration in the longitudinal direction
- Diffusion in all directions.
- Chemical reactions and wood swelling
- Chemical modification of wood. Alkali diffusion coefficient is changed

**Issues not considered in the literature**
Chips treated with alkali shows a impregnation front

The size of the intact core reduces with time.

Situation depends on the wood and treatment conditions.
Alkali impregnation of hardwood in transverse direction

For poplar (Zanuttini et al., JPPS 2003) and for eucalyptus wood (Zanuttini et al., Holz un Roh, 2003), it was shown:

At temperatures below 100 °C, impregnation is a reactive diffusion process.
Consumption could be as high as 6.0 % NaOH / wood

- Deacetylation is the main reaction and is the main responsible for alkali consumption.
Mechanism

At low temperature, it was shown that:

During impregnation, a reactive front is established which separates an intact inner zone from a reacted and swollen outer zone.
Pre-steaming and pressurized impregnation

- Presteaming rapidly heats the wood and the steam produced inside chips is useful to displace the air (Malkov et al. 2000).

- When chip is immersed in liquid under pressure, a high liquid uptake takes place (Malkov et al. 2000).

- Presteaming stage and pressurized impregnation should be considered for a impregnation analysis.
In this paper:
The pattern of the pressurized alkaline impregnation is analyzed:

Profiles of:

- alkali concentration
- alkali content
- liquid content
- acetyls content

It is shown that the level of impregnation can be predicted for given operation conditions
Experimental
Cubes from eucalyptus wood

Cubes

Outer Zone

Inner Zone
Impregnation experiences

A Weverk digester:

• Steaming of wood samples by the liquor vapor
• Pressurization by Nitrogen
Positions of Weverk digester during impregnation

Wood Samples

N₂

Pressure relief

(1)  (2)
Procedure stages

1) Preheating of liquor at 100 °C
2) Load of samples (under the digester cover)
3) Heating, air relief and steaming
4) Immersion in the liquor (Position 2) and pressurization up to 6 Bar (N₂)
5) Treatment under digester rotation
6) Relief of digester (wood submerged in the liquid). Flash in wood was avoided (Position 1)
7) Opening of digester and immersion of cubes in liquid Nitrogen by 2 hours.
8) Store at freezer temperature.
Slicing of treated cubes

Wood sample is still frozen

Slices are weighed, and chemical analyzed
Tangential face of impregnated wood

Wood vessels

1 cm
Chemical analysis of slices
  • Titration
  • Drying
  • Acetyl content by GC chromatography
Results
Results

• For Eucalyptus and Pine

• Profiles in radial direction

as a function of the position from de interphase

• Liquid content of the internal zone of the wood
Eucalyptus – 15 minutes

- **ALKALI (g NaOH / L)**
- **ALKALI (g NaOH / kg)**
- **ACETYLS (x10)**
- **LIQUID (x5 g water/g)**
- **REACTION ZONE**

**Graph:**
- **Position from the Surface (mm):** 0.0 to 2.0
- **Outer Zone:** ALKALI and ACETYLS
- **Reaction Zone:** ALKALI and LIQUID
- **Inner Zone:** ACETYLS

**Legend:**
- Red square: ALKALI (g NaOH / L)
- Black triangle: ALKALI (g NaOH / kg)
- Blue square: ACETYLS (x10)
- Black diamond: LIQUID (x5 g water/g)
- Dashed: REACTION ZONE
Eucalyptus – 30 minutes

- ALKALI (g NaOH / L)
- ALKALI (g NaOH / kg)
- ACETYLS (x10)
- LIQUID (x5 g water/g)
- REACTION ZONE

POSITION FROM THE SURFACE (mm)
Latewood of pine

![Diagram showing changes in alkali and acetyl groups across the reaction zone of latewood of pine.](image)

- **ALKALI** (g NaOH / L)
- **ACETYLS** (x10)
- **LIQUID** (x5 g water/g)
- **REACTION ZONE**

**Axes:**
- **Position from the surface (mm)**
- **Alkali** (g NaOH / kg)
- **Liquid** (x5 g water/g)

**Zones:**
- **Outer Zone**
- **Reaction Zone**
- **Inner Zone**
Mechanism of the chip impregnation

- Liquor diffusion and reaction
- Spent liquor penetration
- Liquor penetration and reaction
## Liquid content inside wood

<table>
<thead>
<tr>
<th>Liquid Content (g / g wood)</th>
<th>Eucalyptus grandis</th>
<th>Pinus elliotti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exterior</td>
<td>1.3</td>
<td>Early-Wood</td>
</tr>
<tr>
<td>Central</td>
<td>0.75</td>
<td>Late-wood</td>
</tr>
<tr>
<td>Central</td>
<td>2.5</td>
<td></td>
</tr>
<tr>
<td>Exterior</td>
<td>0.7</td>
<td></td>
</tr>
</tbody>
</table>

**Pinus elliotti**

- Early-Wood: 2.5
- Late-wood: 0.7

**Eucalyptus grandis**

- Exterior: 1.3
- Central: 0.75
Prediction of the impregnation level

The chip half-thickness distribution

Fraction of impregnated wood
Prediction

If the speed of moving front is known, the level of impregnation can be predicted.
Conclusions (1)

- Steaming (5 minutes) and pressurized immersion (6 Bar) can complete the liquid penetration.
- Initially alkali does not reach the core a chip.
- The chemical impregnation: reaction and diffusion.
  A front of the impregnation is always established.
Conclusion (2)

The time needed for chemical impregnation (time in which the front reaches the center of the chip) can be predicted for a given wood and treatment conditions.
The end