ADVANCES IN EUCALYPTUS PULP BLEACHING TECHNOLOGY

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ABSTRACT

Pulping and bleaching of eucalyptus is subject to intensive research. The goal is to improve the pulp quality, decrease discharges and reduce capital investment. Today pulp of 92+ISO brightness can be produced at low chemical consumptions, less than 25 kg act Cl/adt. There are several options for bleaching sequence. Oxygen, ozone, peroxide and chlorine dioxide can be used in different ways. The removal of HexA has been adopted as an essential part of eucalyptus bleaching.

Pulp washing in bleaching has come to play an important role. When chemical consumptions are lowered, the carry-over amounts in each bleach stage must be maintained as low as possible. This feature is emphasized with reduced use of fresh water.

Today the capacity of a modern single-line mill exceeds 1 million tons of pulp annually, and the trend seems to be only growing. This means that there is a continuous need for the development of new machineries and solutions to meet this demand.

INTRODUCTION

The 1980’s saw an intense increase in the use of eucalyptus as raw material for pulp. We then learned about the use of short-fibred pulp and its advantages in paper manufacturing and witnessed a dramatic increase in its demand. Today eucalyptus pulp is the most desirable raw material for fine paper. This is thanks to its good fiber properties and, above all, its uniform quality. Approximately 15 million tons of eucalyptus pulp is produced annually today, and this quantity is rapidly increasing. The increase in the pulp production capacity has been in the southern hemisphere. In this area the fast-wood plantations produce advantageous pulp raw material of uniform quality and in the vicinity of the production plants. The circulation speed of these fast-wood plantations is between six and eight years, and consequently the wood supply areas need not be large. Hence, to lower the investments costs, efforts are made to dimension the mills’ production capacities as high as possible in single-line implementations. At this moment, the maximum size is 1-1.2 million t/a and the trend seems to be only growing. There is no seasonal variation in the wooden raw material as in the northern hemisphere and hence the maximum pulp production potential can be taken out of the mill equipment on a continuous basis.

The wooden raw material from the plantations is of uniform quality and this quality is continuously enhanced. Good care is taken of the plantations, which are under strict control. A new generation of trees has been developed, superior to previous generations. Numerous eucalyptus species have been developed for the use of pulp mills. Eucalyptus chips normally delignify uniformly and the pulp brightens easily. However, there are wood species that differ from each other in terms of pulping, brightness development and drainage properties. For this reason, the raw material used by the mills must always be considered in the selection of process technical concepts and in the dimensioning of the equipment.

BLEACHING PROCESS

Only a couple of decades ago pulp bleaching was mainly based on the use of chlorine and a minor amount of chlorine dioxide. The sequences normally included five or more bleaching stages. A little later brownstock oxygen delignification, the oxygen reinforced alkali stage, and peroxide as a bleaching agent were adopted in bleaching. Sequence chlorination was developed to solve the pulp quality problem caused by increasing process closure and elevated pulp temperature in chlorine bleaching. Kappa number before bleaching was between 14 and 18 and the total active chlorine consumption up to 60-70 kg/ad. Pulp brightness was typically between 88 and 90 ISO. Since then there has been a dramatic decrease in chemical consumptions (Fig.1).

Bleach line production capacities were limited to 1500 adt/d due to limited equipment capabilities. At the beginning of the 1980’s, medium consistency technology was developed and in many respects this technology opened new opportunities in fiberline unit operations and development. Medium consistency pumping enabled an increase in the pumping volume and
head, and high shear mixing improved the bleaching chemical homogeneity in the pulp. Vacuum washers were commonly used for pulp washing in bleaching. Diffuser bleaching and displacement bleaching systems were also developed. This period when the efficiency of pulp washing and chemical mixing became increasingly important passed by quickly. Fresh water was mainly used for pulp washing together with a split or jump flow principle of filtrates. Water consumption and effluent volumes from bleaching have decreased from 40 to below 15 m³/adt.

At the end of the 1980’s pulp mill effluents and organic chlorine compounds in effluents became a hot topic. This was followed by an intensive bleaching research period. The first target was to move away from the use of elemental chlorine to the 100% use of chlorine dioxide. This bleaching technique was called ECF bleaching. The next target was to also take out chlorine dioxide from the mill process and move to TCF bleaching technology. Thanks to research and development activities, oxygen delignification was enhanced, peroxide bleaching was intensified and the ozone bleaching technology was industrialized at the beginning of the 1990’s. Some mills introduced the enzymatic pre-treatment of pulp. Peracetic acid (P₄A₃) and molybdate (P₄Mo) can be used as well, especially in TCF bleaching.

The development work that was conducted made it possible to bleach kraft pulp to full brightness using TCF bleaching. The total production economy of fully bleached pulp with TCF technology does not yet meet the total economy of ECF technology. However, the use of oxygen, peroxide and ozone in modern ECF bleaching sequences has been established. These types of sequences are called light ECF sequences. Thanks to the abandonment of chlorine and the new way of using peroxide and oxygen, the AOX in bleach effluent has been reduced dramatically (Fig. 2).

**Fig. 1. Active chlorine consumption in eucalyptus bleaching. Brightness target level 90 ISO**

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Recent topics in pulp bleaching technology

Often discussed topics in the bleaching of pulp are pulp quality, the consumption of utilities such as chemicals, water, steam, and electricity, and the quantity and quality of effluents.

Recent research has demonstrated that from the point of view of chemical consumptions and pulp brightness reversion it is important to wash the pulp to a low COD content prior to bleaching. Each kilogram of COD leads to an as high as 0.8...0.9 kg/adt increase in active chlorine /15/. Additionally the COD contained in the wash water for pulp after the oxygen stage can be detrimental.

Oxygen delignification is performed in one or two stages. Both systems may lead to the same average kappa reduction. The two-stage system produces a more uniform final result and lower kappa deviation, enabling a higher kappa reduction. Especially important is pulp brightness after the oxygen stage. High pulp cleanliness prior to the oxygen stage (COD below 90 kg/adt) creates the prerequisites for efficient delignification. By means of the oxygen stage parameters (chemical dosage, temperature, retention time) it is possible to alter brightness up to 5-10 ISO units. This brightness advantage remains all through bleaching. A large proportion of the kappa number of eucalyptus pulp originates in HexA, which cannot be removed in oxygen delignification.
A recent topic of high interest is HexA, its removal from the pulp and effect on bleaching technology.

**Removal of hexenuronic acid**

It has long been known that not even two-stage oxygen delignification is enough to lower the kappa number of eucalyptus or hardwood pulps in general as much as of softwood pulps. Additionally, ECF bleached eucalyptus pulp has a high brightness reversion. A solution to these problems was found after the discovery of hexenuronic acid.

In 1995 a research team from the State Institute for Technical Research (VTT) and the Helsinki University of Technology (HUT) discovered hexenuronic acid, HexA. All pulps contain HexA after the cook /1/. The HexA contents in softwood and hardwood pulps are 20...30 and 40...70 mmol/kg respectively, in eucalyptus pulps even up to 85 mmol/kg. HexA was found to consume permanganate and consequently also bleaching chemicals, chlorine, chlorite dioxide, ozone, and peracetic acid. Approximately 10 mmol/kg of HexA corresponds to one kappa number (Fig. 3). HexA does not react with oxygen or peroxide, which explained the lower kappa reduction of hardwood pulps in oxygen delignification, and the higher kappa in peroxide bleached pulp. It was also discovered that HexA in pulp has a yellowing tendency caused by time, heat and light.

Scientists found that the HexA could be removed from the pulp with the help of mild acid hydrolysis.

pH 3...3.5  
T 85...95°C  
t 120...180 min

With these conditions, 60...75% of the HexA can be removed. This means a kappa reduction of 3...5 units, which results in significant bleaching chemical savings. Figure 4 shows the HexA removal as a function of retention time at different temperatures /2/. The simulated result corresponds very well to mill experiences. Kappa reduction of 5 units (50 mmol/kg) has been achieved with a retention time of 2 hours at a temperature of 95°C /9/.

**Fig. 4. HexA removal as a function of time at different temperatures.**

The first industrial HexA removal stage, the A-stage, was taken into use in 1997. It was conducted in the storage tower. The first whole bleaching sequence where an A-stage was combined with bleaching was taken into use at Aracruz Pulp Mill in 2002 with excellent results /16/.

HexA and lignin react with chlorine dioxide at the same speed (Fig 5) /1/. Ozone and peracetic acid prefer to react with HexA instead of lignin. HexA removal before ozone bleaching always leads to a lower kappa reduction per used ozone.

**Fig. 3. Relation between HexA and residual lignin in eucalyptus pulp.**

When understanding the role of HexA, the delignification degree in the oxygen delignification stage is the same as for softwood and hardwood even though the kappa reduction is very different.
eucalyptus pulp is, for example, A/Do-Eop-D(-P). When the target brightness is ISO 92+ it is advantageous to introduce a peroxide stage as the fourth bleaching stage. This is also important when reducing the OX content and improving the brightness stability of bleached pulp. In alternative applications HexA removal is implemented in a long hot Do-stage called the D_{Hot-stage} /13/. The retention time is typically 2 hrs. Heating of pulp and the charge of chlorine dioxide are done before the treatment vessel. Nevertheless, the target is to remove HexA even though dioxide is present.

It has also been proven that HexA is involved in oxalic acid formation; oxalic acid is formed especially in ozone reactions. HexA can in itself bind harmful metal ions like Ca, Mn and Fe. The decomposition of HexA reduces oxalic acid formation and the binding locations of metal ions in pulp, hence reducing – and even eliminating – potential metal compound scaling in process equipment.

**Modern bleaching sequences**

ECF bleaching is a dominant concept, but there are several variations in the way of building the bleaching sequence. In specifying the bleaching sequence, various factors such as target brightness, the chemicals to be used, costs and emissions must be kept in mind. With eucalyptus pulp, the basic starting point is almost invariably a high HexA content. Preferably the bleaching sequence should be started with the removal of HexA. With a brightness target of 91-92+ ISO, four-stage bleaching is the most advantageous alternative in terms of operating costs, in spite of the fact that the investment in equipment is higher than for bleaching in three stages. Ozone is a very efficient bleaching chemical, alone or combined with chlorine dioxide. Its advantageousness must be assessed separately in each case taking the energy price and pulp quality requirements into account. As an example, different sequence alternatives and an evaluation of them compared to four-stage bleaching A/D-Eop-DnD including three intermediate washers are shown in table 1. All the sequences are considered to achieve 90 ISO brightness.

![Fig. 5. Competing removal of HexA and lignin.](image)

In the hydrolysis, HexA decomposes to furan carboxyl acid, FCA. FCA does not in practice react with ClO₂ (Fig.6). However, in the D-stage a small amount of hypochlorous acid is present, which reacts with FCA, lignin and carbohydrates. In the presence of lignin containing material, chlorine dioxide prefers to react with lignin instead of reacting with FCA. Since the discovery of HexA it has been known that ozone rapidly reacts with FCA /1,3/.

![Fig. 6. Reaction of 2-furoic acid with ClO₂ and HOCl/Cl₂ (T=90°C, pH=2, c[2-furoic acid]=0.086 mmol/l, c[NaOCl]=0.69 mmol/l aCl/l, c[ClO₂]=4.30 mmol/l). The change in absorbance of 2-furoic acid was recorded at 245 nm.](image)

This discovery led to the use of the A-stage combined with the first dioxide stage without interstage washing. When the A-stage is combined with an ozone stage, interstage washing is needed. The removal of HexA is becoming established in eucalyptus pulp bleaching. A modern bleaching sequence of...
Table 1. Relative benefits (+/-) of different bleaching sequences to each other.

<table>
<thead>
<tr>
<th>Sequence</th>
<th>Capital cost</th>
<th>Chemical cost</th>
<th>Brightness</th>
<th>Brightness</th>
<th>Brightness</th>
<th>Effluent</th>
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Several other sequences are also possible; especially in the modernization of existing old bleach processes. An interesting sequence is Z-Eop-D/P, for example. The filtrate loop can be coupled to the recovery cycle in principle, but the additional sulfur and sodium must be taken into account in the recovery. However, carry-over buildup lowers process efficiency and interest in industry has not been very high.

Different investigations have revealed that bleaching reactions in the chlorine dioxide stage occur very rapidly, in 5 to 10 minutes, when the temperature exceeds 65°C. When combining the Do-stage with the A-stage, the Do-stage can be done in a couple of minutes. Recent investigations have shown that a short retention improves brightness stability.

In some applications A/Do stages are combined into one and the same stage called the D\textsubscript{hot} stage. The A/Do sequence has been found more selective than the D\textsubscript{hot} stage. Chemical savings always depend on the pulp to be bleached. For the hot Do stage, all process equipment must be built to tolerate chlorine dioxide conditions.

Washing in bleaching

Recent mill experiences have shown that pulp washing is very important in terms of the end result of bleaching. Especially important is pulp cleanliness in the first stage of bleaching. The uncleanliness may originate in oxygen stage washing or consist of circulating bleaching impurities. Research has shown that the COD that comes to bleaching together with the pulp and the COD circulating in bleaching have a detrimental effect. The effect of the COD originating in the Do stage is especially detrimental. It increases the chemical consumption and deteriorates brightness development and may even stop it.

Figure 7 presents the effect of the D\textsubscript{100} stage and post O\textsubscript{2} stage filtrates on the result of the D\textsubscript{100}-Eop sequence /4/. The results show that the COD affects both kappa number and brightness. In this case, the additions of 10 kg/adt COD from the post O\textsubscript{2} stage had an influence of one brightness unit. The addition of 8.5 kg/t COD from the D\textsubscript{100} stage, on the other hand, decreased the brightness by more than 3 units.

![Fig. 7. Effect of post O\textsubscript{2} carry-over and D\textsubscript{100} recycle to D\textsubscript{100} stage on D\textsubscript{100}-Eop kappa number and brightness. The letters PO stand for post oxygen.](image-url)

Figures 8 and 9 present the kappa number and brightness after Do-E pre-bleaching /5/. COD from different sources is added to the Do stage. COD from cooking has the most detrimental effect on both kappa number and brightness. When pulp is diluted with Do filtrate after the post-O\textsubscript{2} washer, the D0 COD input into the Do stage is typically between 8 and 12 kg/adt. As the figures show, the impact on the E-kappa number and brightness is 0.5…0.8 and 2.5…3.6 ISO respectively with the same chemical dosage.
The higher kappa number and lower brightness after the E stage must be compensated by a higher bleaching chemical dosage. However, other tests have proven that circulating carryover may cause a brightness ceiling. In fig. 10 the D-E-D sequence ended in a brightness of 86 ISO in a clean system but never reached the same brightness even though one more bleach stage and chemical dosage were added /6/.

How then shall the water consumption in bleaching be lowered if the consequence is a clear increase in the consumption of bleaching chemicals and a brightness ceiling? The solution is fractional washing. The first stage of bleaching is maintained clean and the filtrates in the subsequent stages are circulated fractionally. The COD level of the bleaching stages will increase to some extent, but this can be compensated through a slight increase in the chemical dosage and the brightness ceiling can be prevented. An example is shown in figure 11. Total COD output in bleach effluent is 20-28 kg/adt depending on brightness target and the closure of the filtrate system, among others.
Pulp quality

Efforts are made to maintain the raw material fiber potential in the pulp production process. The basis for the pulp strength is generated in the cook. This strength can be destroyed in the systems after the cook, but there is no chance to improve it. Normal strength delivery in bleaching is close to 100%. New process technology must always face prejudice; this has been the case also for ozone bleaching and the removal of HexA. However, by optimizing the process conditions for the raw material concerned it has been possible to prove the suspicions unjustified.

The physical properties of pulp are a combination of the properties of individual fibers. The focus of recent research is on fiber level phenomena. Fiber deformation or damage can be caused by a chemical or mechanical attack. Elevated temperature and alkalinity expose the fibers to mechanical deformation in the equipment. Mechanical treatments like pressing and disintegration may result in fiber damage /10, 11/. From this point of view the conditions in bleaching are not very critical, but the trend is towards less equipment.

An important quality factor especially for a market pulp mill is brightness stability. There are many factors that affect brightness stability. Pulp lignin content is known to reduce brightness. Pulp HexA content has also been found to affect brightness stability /14/. Figure 12 shows brightness reversion in laboratory-bleached eucalyptus pulps. Brightness target has been 90 ISO and reversion is measured after 4 hrs dry aging at 105°C. Experiences have shown that below the HexA of 5 mmol/kg there is no clear impact on reversion.

Recent research has shown that the amount of carbonyl groups in pulp strongly affects brightness stability. Carbonyl groups are generated in reactions between hypochlorous acid and cellulose, and hemicellulose. Hypochlorous acid is present in the dioxide stage depending on the chemical dosage and pH. Figures 13 and 14 show that there is a correlation between active chlorine and carbonyl groups, and between carbonyl groups and brightness reversion /7/.

An important quality factor especially for a market pulp mill is brightness stability. There are many factors that affect brightness stability. Pulp lignin content is known to reduce brightness. Pulp HexA content has also been found to affect brightness stability /14/. Figure 12 shows brightness reversion in laboratory-bleached eucalyptus pulps. Brightness target has been 90 ISO and reversion is measured after 4 hrs dry aging at 105°C. Experiences have shown that below the HexA of 5 mmol/kg there is no clear impact on reversion.

For the paper manufacturer, the OX content in bleached pulp is an important quality parameter. It is mainly the used amount of active chlorine that has an effect on the generation of OX. The removal of HexA clearly lowers the OX content of the pulp, as does the ending of bleaching at the peroxide stage (Fig 15) /8/. Final brightness of all eucalyptus pulp samples has been 90%
ISO. In the sequence of DEDD, the lower pH in the last D stage increases the OX content (pH 3.8 vs. 3.5). The higher chlorine dioxide charge in the Do stage also increases the OX content in the A/DEDD sequence (10 vs. 20 kg act Cl/ad t). A final P-stage further decreases the OX in the pulp.

![Fig. 15. OX in pulp in different bleaching sequences.](image)

**Results**

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<th>Sequence</th>
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**Power, steam and effluent treatment**

Total power consumption in bleaching is the sum of the power consumed for the pumping of pulp and filtrates and the use of equipment. Power consumption per ton of pulp has decreased due to the increase in size of production lines and equipment. This is true also for pumping, where the pulp consistency has increased and the amount of circulating liquid has decreased. Beside the regulation of valves, variable speed motors are commonly used. Pressurized reactor stages require feed pumps with a large motor size. That is why atmospheric towers should be preferred. The power consumption of modern 4-stage bleaching is 70-80 kWh/ad t.

Steam is consumed for the heating of bleaching stages. Today direct steam heating is used in the oxygen stage only; in bleaching heating occurs indirectly by heating filtrates and washing liquids.

Efforts are made to minimize steam pressure to achieve the maximum power production. The practical minimum for steam pressure is approximately 9 bars. Low steam pressure leads to booster pumping between the oxygen reactors.

Steam consumption in new bleach plants has decreased due to lower water consumption and the closure of filtrate circulations. In a modern eucalyptus bleach plant, steam consumption is on the level of 350-450 kg/ad t. The effluent temperature of a bleach plant with closed filtrate circulations is 80-85°C. Prior to effluent treatment, the filtrates must be cooled, but the heat can be partly utilized inside the bleach plant.

The largest proportion of the pulp mill effluents originates in bleaching. In new mills, the volume of effluents from bleaching is 10-15 m³/ad t. Figure 16 shows a diagram of a modern biological effluent treatment system. The process includes an effluent primary clarifier, a neutralization tank and a cooler prior to biological treatment. In the secondary clarifier, the sludge of the biological treatment process is separated. The wastewater can additionally be directed through a tertiary treatment stage, where the color can be reduced. Typical reduction degrees are:

- COD 70-75%
- AOX 50-60%
- BOD5 95%
- Color 60-70%

![Fig. 16. A modern effluent treatment system](image)

**Recent mill results of eucalyptus bleaching**

**Veracel results**

The Veracel pulp mill has now been running for almost two years. This mill has proven that with
intensive training, good co-operation with suppliers and a strong organization, an unprecedented learning curve and design capacity can be reached. Excellent chemical consumption figures and pulp quality parameters have been achieved.

Another area will be environmental parameters in the effluent. The bleach sequences have been renewed and the effluent parameters may need to be reevaluated. Closure of the filtrate loops inside the bleach plant is a reality today but recycling to recovery is still an open question. Recycling of filtrates leads rather invariably to disadvantages in process economy and pulp quality. Separate treatment of bleach filtrates by means of evaporation or other techniques has not yet broken through.

A great deal of development work has already been done in the modeling and simulation area. Bleach reactions in each bleach stage and soon through the whole bleach process can be modeled and simulated /12/. This may enable the development and optimization of bleach process systems with much less laboratory work.

CONCLUSIONS

- Double-stage oxygen delignification creates the basis for low chemical bleaching.
- Low carry-over to bleaching and a low carry-over amount in each bleach stage is very important.
- ECF bleaching is a dominant bleach concept. Several variations of bleach sequences are possible.
- HexA removal is an established stage in the bleaching sequence. A four-stage sequence A/D-Eop-D-P is recommended for 92+ISO brightness.
- Brightness reversion is improved through the removal of HexA. Reduction of act. chlorine usage and a final P-stage improve brightness stability.
- OX in pulp is reduced by lower act. chlorine usage and a final P-stage.
- Electricity and steam consumption in a modern bleach plant is 70-80 kWh/adt and 359-450 kg/adt respectively.
- Effluent flow from ECF bleaching is 10-15 m³/adt. AOX, COD and BOD5 discharges are very low after a modern effluent treatment system.
- A chemical consumption of 23 kg act. Cl/adt and 5 kg/adt peroxide in four-stage bleaching to 92.5 ISO in mill conditions has been achieved.
REFERENCES


Development of HexA as a function of time (pH = 3.5)

Fig. 4. HexA removal as a function of time at different temperatures.

<table>
<thead>
<tr>
<th>Sequence</th>
<th>Capital cost</th>
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<th>Brightness flexibility</th>
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Table 1. Relative benefits (+/-) of different bleaching sequences to each other.
Fig. 8 Effect of COD originating from different sources on Kappa number increase after the E-stage.

Fig. 9. Effect of COD originating from different sources on brightness decrease after the E-stage.
Fig. 11. 3-stage bleach plant with fractional and non-fractional washing at normal and reduced effluent discharge.

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<td>8.9</td>
<td>5</td>
<td>1.9</td>
</tr>
<tr>
<td>Bleach Plant Effluent $10.8 \text{ m}^3/\text{adt}$</td>
<td>5</td>
<td>22.8</td>
<td>14.5</td>
<td>3.6</td>
</tr>
<tr>
<td>Segregated washing</td>
<td>17.9</td>
<td>16.6</td>
<td>11.5</td>
<td>5.4</td>
</tr>
<tr>
<td>Non-segregated washing</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Fig. 16. A modern effluent treatment system
Fig. 17. Veracel Fiberline