TCF Bleaching of Eucalyptus Kraft Pulp: The Selection of the Sequence and the Best Conditions

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Bleaching was conducted with conventionally oxygen-delignified pulp. The residual lignin was lowered either with an OAqZO or an OAqPeracidO treatment. Kappa numbers between 3 and 5 were achieved without any viscosity penalty. The bleachability was better after ozone activation. A concentration of 0.25% ozone was more effective than 1.5% peracetic acid. Final brightness was above 90% ISO at similar strength properties. Final bleaching with hydrogen peroxide was made with only one stage. The addition of magnesium sulphate and sodium silicate improved the results. The lowest hydrogen peroxide demand resulted at temperatures below 100°C with a retention time of up to 24 h. The 90% ISO brightness level needed an input of less than 2% H_2O_2. *Bleaching at 110°C accelerates the reaction, but yields inferior results in terms of peroxide consumption and viscosity.*

INTRODUCTION

The conversion of conventional bleaching sequences for sulphite pulps to TCF sequences was a rather rapid process. The good bleachability and the small number of bleaching stages required made the implementation technically very simple. The modification of existing bleaching stages was inexpensive and the results were good. The installation of high-consistency bleaching stages for the final hydrogen peroxide treatment allowed a significant decrease in bleach chemical demand. Today, most of the mills with modified equipment have bleaching costs equal to or below the costs of chlorine bleaching. They have no AOX or OX loads in the effluent and no storage or handling problems with hazardous chlorine-containing chemicals.

In contrast to this development, the current practice for TCF bleaching of kraft pulps is far from being optimized. Currently, only a small number of bleaching lines for TCF bleaching of kraft pulp have been constructed. As a consequence, the results in terms of brightness increase and bleaching chemicals consumption are not always satisfying. It is the aim of this paper to describe the optimization of a TCF bleaching sequence. In order not to narrow the applicability of the bleaching sequence discussed, the starting points were defined as a) conventional continuous pulping and b) conventional oxygen delignification. Typically, the kappa numbers achieved during conventional pulping of eucalyptus wood are between 17 and 15. Conventional oxygen delignification decreases the residual lignin level to about kappa 10. A pulp from an existing pulping line was used as the raw material.

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TRANSITION METALS REMOVAL

The key to good results in TCF bleaching is the elimination of transition metal ions from the pulp [1] because these interfere with the bleaching action of peroxygen compounds. In kraft pulping, unlike sulphite pulping, the metal ions are not water soluble after the pulping process. During pulping, metal ions get reduced to a low state of oxidation and are precipitated as sulphides. These sulphides are very insoluble under alkaline and neutral conditions, so normal washing does not remove them. Oxygen delignification might oxidize them to a higher oxidation state, but the resulting hydroxides are still insoluble under the conditions of oxygen-stage washing. They become water soluble under mild to strong acidic conditions. Table I shows the removal of metals from an eucalyptus pulp. The pulp used for the tests had a rather low content of iron and manganese. A part of the residual metal seems to be bound to the lignin or to wood polyoses [2] and becomes accessible only with increasing delignification.

ACTIVATION OF A SECOND OXYGEN STAGE

Brightness increase is a function of the residual lignin level. To decrease the lignin content to a level at which peroxide can bleach effectively, an additional step is required beyond the first oxygen stage. The next treatment step could either be a second oxygen stage, or it could be an activation step for the subsequent oxygen treatment. In the first alternative, the oxygen reaction can be boosted by raising the temperature above the boiling point. On top of this, delignification can be improved with the addition of

TABLE I
REMOVAL OF TRANSITION METALS FROM OXYGEN-DELIGNIFIED EUCALYPTUS KRAFT PULP. CONDITIONS: 3% CONSISTENCY, 60°C, 0.5 h, 1% H_2SO_4**, 0.5%** $NaHSO_3$**, pH 2.3**

	Fe (ppm)	Mn (ppm)
oxygen delignified	25	5
A	11	<1
Aq with 0.1% EDTA	10	<1

TABLE II
DELIGNIFICATION OF EUCALYPTUS KRAFT PULP WITH OXYGEN AND HYDROGEN PEROXIDE AFTER OXYGEN TREATMENT TO KAPPA 10 AND ACID WASH. CONDITIONS: 2% NaOH, 0.1% $MgSO_4$, 10% CONS., 1.5 h, 0.3 MPa O_2

stage	temperature (°C)	H_2O_2 addition (%)	kappa #	brightness (%ISO)	viscosity (mPa·s)
O	90	–	8.4	58.2	22.1
O	110	–	6.5	64.8	21.7
Op	110	2	6.0	82.2	17.1

TABLE III
DELIGNIFICATION OF OAq-PRETREATED EUCALYPTUS KRAFT PULP WITH PERACIDS AND OXYGEN. PRETREATMENT AT 10% CONS., 70°C, 1 h, pH_{end} 4.2 TO 3.5. OXYGEN STAGE WITH 2% NaOH, 0.1% $MgSO_4$, 0.3 MPa O_2 AT 110°C, 1.5 h, 10% CONS.

sequence	peracetic acid (%)	Caro's acid (%)	oxygen-stage results kappa #	viscosity (mPa·s)
-PaaO	0.75		5.5	21.6
-PaaO	1		4.7	21.4
-PaaO	1.5		4.1	20.6
-CaaO		1.5	4.3	19.1
-O (see Table II)	–	–	6.5	21.7

TABLE IV
EFFECTS OF OZONATION AND SUBSEQUENT OXYGEN TREATMENT. OZONATION AT 30% CONS., pH 4 TO 5, AMBIENT TEMP.; OXYGEN STAGE WITH 2% NaOH, 0.1% $MgSO_4$, 0.3 MPa O_2 AT 110°C, 1.5 h, 10% CONS.

sequence	ozone (%)	oxygen stage results kappa #	brightness (% ISO)	viscosity (mPa·s)
-ZO	0.2	5.8	69.4	18.8
-ZO	0.25	4.9	72.4	17.1
-ZO	0.5	2.3	75.3	12.5
-O (see Table II)	–	6.5	64.8	21.7

hydrogen peroxide. This is today's most widely practiced method to lower the lignin residual in TCF bleaching. Results of a second oxygen stage with and without hydrogen peroxide addition are given in Table II.

The addition of hydrogen peroxide produces a significant increase of the brightness, but only a moderate drop of the kappa number. Obviously, the high temperature results in increased thermal decomposition of hydrogen peroxide, as shown by the drop in viscosity. A kappa number reduction of only half a unit at the expense of 2% of hydrogen peroxide is not a satisfying result. The brightness increase looks promising; however, at kappa 6, final brightening to 90% ISO is not within the reach of a subsequent peroxide stage. Even with the addition of 4% H_2O_2, only 89% ISO brightness was achieved (see Table VI below).

The delignification achieved in the second oxygen stage has to reach lower kappa numbers to allow brightening to 90% ISO. Possibly, an activation step prior to the second oxygen treatment could achieve this. Two types of activation can be considered: treatment with a peracid or with ozone. Both compounds have advantages and disadvantages. Peracids react with the residual lignin as electrophiles under weak acidic conditions. The resulting oxidized lignin is more easily degraded in a subsequent oxygen stage. The performance of peracids is best at temperature levels between 50 and 70°C, which fits into the temperature profile between two oxygen stages. Peracetic acid (Paa) or peroxomonosulphuric acid (Caro's acid, Caa) can be used for the activation [3]. If applied at the same pH, both compounds yield nearly identical results. Both peracids have to be produced on site, either by mixing sulphuric acid with hydrogen peroxide, or via vacuum distillation of peracetic acid from the equilibrium formed between acetic acid, hydrogen peroxide, water and the peracid. The results of activation with peracid and subsequent oxygen delignification are summarized in Table III.

Peracetic acid produces slightly higher viscosity values compared with Caro's acid. To achieve a visible drop in lignin level, about 1% peracid is required. The ratio of 2 to 3 for peracetic acid to Caro's acid takes the difference in molecular weight into account.

The alternative to peracid activation is pretreatment with ozone. Ozone is a very reactive compound and has to be mixed thoroughly with the pulp to avoid local overoxidation. In consequence, the amount of ozone that can be applied without cellulose degradation is limited. Ozonation needs lower temperatures compared with peracid treatment. To avoid fibre damage, the ozonation temperature should not be higher than 40 to 50°C. With the ozone treatment sandwiched between the hot oxygen stages, it could be difficult to run a medium-consistency stage at this temperature level, especially with tight water recycle. High-consistency ozonation is easy to accomplish on a laboratory scale. A fluidized bed reactor is used in our laboratory for homogeneous mixing [4]. The effect of ozonation on the degree of lignin removal in subsequent oxygen delignification is listed in Table IV.

Ozone is significantly more effective compared with peracids. The amount of ozone needed to achieve kappa numbers below 5 is 0.25% or higher. However, the ozone charge of 0.5% had a significant negative effect on viscosity.

PEROXIDE BLEACHING

The success of peroxide bleaching depends on the level of residual lignin. In addition, bleachability is affected by the delignification method. It is not possible to simply specify a kappa number that is low enough to allow bleaching to the target brightness. On the other hand, the lower the lignin level, the easier final bleaching becomes. In order to minimize the hydrogen peroxide requirement, the following parameters are of importance: stabilization, buffering, temperature, time and consistency.

The initial reaction of hydrogen peroxide with pulp is very fast. This phase is followed by a pronounced slowdown of the brightness increase and the peroxide consumption. This indicates a rapid reaction with compounds that are easily oxidized, and a second phase that needs considerably more effort for the breakdown of the chromophores. The standard procedure to speed up a reaction is to increase the temperature and the concentration. High-temperature bleaching or high-consistency treatment therefore are the standard approaches to accelerate the bleaching reaction. Temperature increase in peroxide bleaching reaches a limit when the thermal decomposition reaction of hydrogen peroxide into water and oxygen begins to dominate over the bleaching reaction. In consequence, very high temperatures may result in a faster consumption of peroxide, but not in a better brightness gain. Stabilization of the bleaching process becomes more important. Small amounts of sodium silicate and magnesium sulphate improve the brightness as well as the viscosity. Table V demonstrates the benefit of the addition of both chemicals.

The advantage of the addition of small amounts of sodium silicate and magnesium sulphate is obvious. Silicate addition primarily results in a higher viscosity level, but has less effect on brightness. In all trials, the residual peroxide was very high.

Table VI compares the results of bleaching with peroxide with and without an additional acid treatment, which was recommended to remove traces of transition metals [5]. Bleaching at 90°C needs a very long reaction time. To complete the reaction, 24 h are required. The residual of hydrogen peroxide in all experiments is within the range that allows one to conclude that the alkalin-

TABLE V
EFFECT OF SODIUM SILICATE AND MAGNESIUM SULPHATE ADDITION IN PEROXIDE BLEACHING. BLEACHING OF KAPPA 6 EUCALYPTUS KRAFT PULP WITH 4% H_2O_2 AT 90°C, 10% CONS., 8 h

trial	NaOH (%)	sodium silicate (%)	$MgSO_4$ (%)	H_2O_2 residual (%)	brightness (%ISO)	viscosity (mPa·s)
1	1.5	–	–	1.22	85.8	11.2
2	1.4	0.5	–	1.71	86.2	14.4
3	1.4	1	–	1.72	86.4	14.6
4	1.5	–	0.1	1.68	86.8	12.3
5	1.5	–	0.2	1.72	86.9	12.5
6	1.4	0.5	0.1	1.88	87.5	14.7

TABLE VI
PEROXIDE BLEACHING AFTER OAqOp DELIGNIFICATION TO KAPPA 6.0 WITH 4% H_2O_2, 1.5% NaOH, 0.5% SODIUM SILICATE, 0.1% $MgSO_4$, AT 10% CONS., 2 h. ACID TREATMENT BEFORE P STAGE WITH 1% H_2SO_4, 0.5% $NaHSO_3$

sequence	temperature (°C)	time (h)	H_2O_2 residual (%)	brightness (%ISO)	viscosity (mPa·s)
-P	90	24	0.10	88.5	11.0
-AP	90	24	0.51	88.9	14.5
-P	110	2	0.27	85.3	11.9
-AP	110	2	0.75	86.3	15.4

ity was neither too low nor too high. The acid wash pretreatment had a positive effect on the brightness development and on the hydrogen peroxide consumption. Peroxide residual and brightness are higher and the viscosity is improved.

The consumption of hydrogen peroxide is accelerated by increasing the temperature to 110°C. Unfortunately, this does not result in a better brightness gain. On the contrary, the faster consumption of the hydrogen peroxide yields a brightness that is about three points lower. Obviously, the temperature increase not only accelerates the bleaching reactions but also the decomposition reactions of hydrogen peroxide. A reason for this decomposition reaction is the low thermal stability of hydrogen peroxide. The activation energy for the cleavage of the oxygen–oxygen bond is rather low (ΔH = –71 kJ mol^{-1}) [6]. This is about one fifth of the energy required to cleave a carbon–carbon bond. Surprisingly enough, this limited thermal stability is not discussed in the papers [7–10] that recommend very high temperature. On the contrary, only transition metals are blamed for causing hydrogen peroxide decomposition. However, metal traces are only one possible source for hydrogen peroxide losses. H_2O_2 can be decomposed by alkali, by temperature and by metal ions [11]. The results of high-temperature experiments run in teflon containers, where the effect of metal surfaces can be excluded, support this interpretation [12]. Our trials were conducted in a laboratory-scale high-shear mixer with injection of the chemicals at the reaction temperature and with immediate mixing. This approach is identical to mill scale, where steam and chemicals are added without a significant time delay. Slow heating to a high final temperature yields better results, but is not comparable to mill practice [11].

An option to increase the reaction rate is the application of higher caustic soda charge. A higher pH level increases the concentration of the perhydroxyl anion. This increase in peroxide consumption is shown in Fig. 1. However, the acceleration of the peroxide consumption is not translated to higher brightness, as shown in Fig. 2. Obviously, the higher pH also accelerates side reactions, such as the alkali-catalyzed decomposition of hydrogen peroxide into water and oxygen. As a consequence, higher alkali charges do not increase the brightness gain.

High-consistency bleaching is another alternative. Table VII gives a comparison of bleaching with a high peroxide charge at three different temperature levels and with high or medium consistency. The pulp was delignified to a lower lignin residual using Caro's acid activation of the second oxygen stage. The results favour neither bleaching at high consistency at 75°C nor the very high temperature reaction at 110°C. This high-temperature treatment was not improved with higher or lower charges of alkali. Compared with medium-consistency bleaching, the advantage of the high-consistency treatment at 90°C was limited. The highest brightness resulted after bleaching at 90°C with 24 h retention time.

COMBINATION OF DELIGNIFICATION AND FINAL BLEACHING

So far, the hydrogen peroxide charge in the bleaching experiments has been rather high. For the economy of the bleaching process, the hydrogen peroxide requirement in final bleaching should be as low as possible. As mentioned above, the bleachability of pulp is a function of the lignin residual, but also depends on the type of treatment. Table VIII gives the bleaching results with H_2O_2 after activation of the second O stage with different charges of ozone and peracetic acid. A rather small amount of ozone is sufficient to achieve a brightness level above 90% ISO. More peracid is required to produce the same degree of delignification, and bleachability is lower.

Figure 3 again compares these results and adds bleaching at high temperature. Peroxide bleaching was conducted either at 90°C with 24 h retention time or at 110°C within 2 h. The bar diagram demonstrates the advantage of the lower bleaching tem-

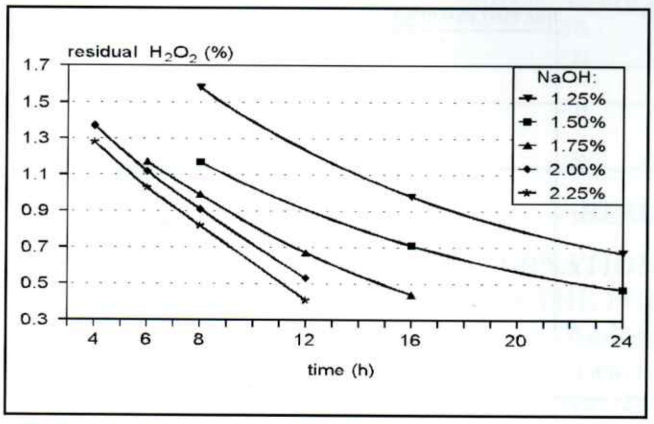

Fig. 1. Effect of increasing caustic soda charge on peroxide residual with time; bleaching with 3% H_2O_2, 0.5 % sodium silicate, 0.1% $MgSO_4$, at 15% cons., 95°C.

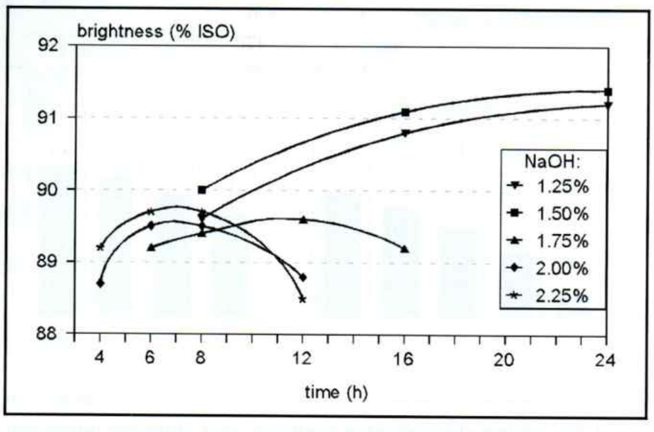

Fig. 2. Effect of increasing caustic soda charge on brightness increase with time, bleaching with 3% H_2O_2, 0.5 % sodium silicate, 0.1% $MgSO_4$, at 15% cons., 95°C.

perature. Both sequences achieve the target brightness of more than 90% ISO. The requirement for hydrogen peroxide is lower at 90°C. The faster reaction at 110°C yields only 88 to 89% ISO with twice the amount of hydrogen peroxide required.

Unfortunately, a tower for 24 h storage in a 1000 t/day mill would be extremely large. A storage capacity of 10 000 m^3 would be required. The very high temperature alternative would need only 1250 m^3 for 2 h retention time. On the other hand, the brightness difference of about three points at the same input of peroxide favours the larger tower. The right compromise seems to be a combination of increased consistency and a temperature level that still accelerates the bleaching process, but does not decompose hydrogen peroxide significantly in side reactions. A consistency of 15% is still within the range of "medium consistency" and, with a retention time of 12 to 16 h, the volume of the peroxide tower would shrink to 4000 to 5000 m^3. Table IX has results of bleaching at 15% consistency with 95 and 100°C.

Figure 4 compares the strength of the pulps from Fig. 3 bleached with 2.5% H_2O_2. The strength properties of the pulps are rather similar. There is no significant difference between the tear or tensile values for the TCF trials with either a peracid or an ozone activation stage. The values achieved in the laboratory tests are close to those from mill data for ECF (ODEopDD) bleached pulp.

These results demonstrate the possibility of producing TCF eucalyptus kraft pulp with very high brightness levels. In contrast to the current practice in most TCF mills, the requirement for the bleaching chemicals ozone and hydrogen peroxide is significantly lower. However, it is necessary to modify the bleaching conditions and use a long retention time in the peroxide stage.

EXPERIMENTAL

Acid wash/chelation was conducted in plastic containers at 3% consistency, 60°C, 0.5 h. Peroxide and peracid stages were carried out in sealed plastic bags at 10, 15 or 25% consistency with variation of time and temperature. Pressurized peroxide stages and Op stages were conducted in a laboratory-scale stainless steel high-shear mixer [13] at 10% consistency with variation of time and temperature. The pulp was preheated to temperature, and the chemicals were injected, followed by mixing for only 15 s. Ozone stages were run in a laboratory-scale fluidized-bed reactor [4] with fluffed pulp at 30% consistency and acidification to pH <5. Brightness and other physical properties were measured according to TAPPI standards. Sequences are abbreviated using the CPPA recommendation.

CONCLUSIONS

The following conclusions can be drawn from the bleaching results:

TABLE VII
PEROXIDE BLEACHING AT 75 OR 90°C AND 110°C; WITH 4% H_2O_2, 0.5% SODIUM SILICATE, 0.1% $MgSO_4$ PULP PREDELIGNIFED WITH THE SEQUENCE OAqCaaOAq TO KAPPA 4.0 (CAA STANDS FOR CARO'S ACID)

temperature (°C)	NaOH (%)	consistency (%)	time (h)	H_2O_2 residual (%)	brightness (%ISO)
75	1.5	25	24	0.91	89.3
			48	0.37	90.5
90	1.5	25	4	1.07	89.1
			6	0.45	90.2
90	1.5	10	8	1.67	89.3
			24	0.74	90.8
110	1.25	10	3	0.42	87.3
110	1.5	10	2	0.68	88.1
			3	0.24	88.5
110	2.25	10	2	0.06	87.5

TABLE VIII
BLEACHING OF EUCALYPTUS KRAFT PULP WITH THE SEQUENCE OAqZOAqP OR OAqPaaOAP. PEROXIDE BLEACHING AT 90°C, 10% CONS., 24 h

ozone (%)	peracetic acid (%)	kappa after 2nd oxygen	H_2O_2 (%)	brightness (% ISO)	viscosity (mPa·s)
0.25		4.9	2	90.6	14.8
0.25		4.9	2.5	91.7	14.7
0.5		2.3	1	89.0	13.7
0.5		2.3	2	90.9	13.2
	0.75	5.5	3	88.4	15.5
	1.5	4.1	2	88.8	19.1
	1.5	4.1	3	90.5	15.0

TABLE IX
OPTIMIZATION OF THE RETENTION TIME/TEMPERATURE RELATIONSHIP IN PEROXIDE BLEACHING. PULP PREDELIGNIFIED TO KAPPA 3.8 WITH THE SEQUENCE OAqZOA; CONST. 2.5% H_2O_2, 1.5% NaOH, 0.5% SODIUM SILICATE, 0.1% $MgSO_4$

temperature (°C)	consistency (%)	NaOH (%)	time (h)	brightness (% ISO)	viscosity (mPa·s)
95	15	1.5	8	90.0	13.9
			16	91.4	13.7
100	15	1.75	8	90.3	13.8
			12	90.5	13.6

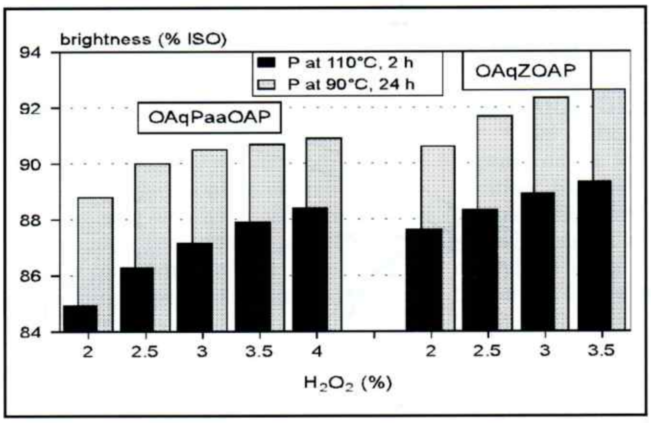

Fig. 3. Bleaching of eucalyptus kraft pulp with different hydrogen peroxide charges at 90°C (24 h) and 110°C (2 h) following OAqPaaOA and OAqZOA treatment. Peracetic acid charge: 1.5%; ozone charge: 0.25%, P stage cons. 10%.

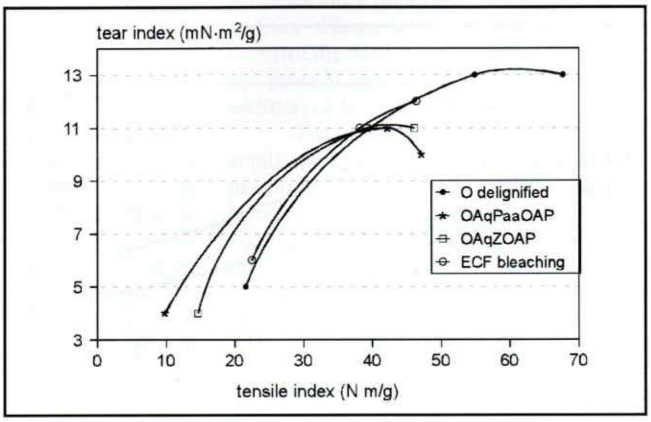

Fig. 4. Strength data of eucalyptus kraft pulp bleached with 2.5% H_2O_2 with the TCF sequences of Fig. 3. Strength of ECF pulp (DEopDD) added for comparison.

- The application of two oxygen stages with metals removal and an activation step sandwiched between the stages produces the degree of delignification to allow final bleaching with one peroxide stage.
- The sequence with the lowest demand for bleaching chemicals is OAqZOAP.
- The requirement for peracid in the OAqPaaOAP sequence is higher than the demand for ozone.
- The strength data of both pulps are very similar.

Hydrogen peroxide bleaching results are best under the following conditions:

- Buffering with small amounts of sodium silicate, addition of $MgSO_4$ and a temperature below 100°C.
- A retention time of about 8 to 16 h is needed.
- High temperature (>100°C) peroxide bleaching is a means to accelerate the bleaching procedure. However, it results in a higher peroxide demand for the same brightness.

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ABSTRACT: Bleaching was conducted with conventionally oxygen-delignified pulp. The residual lignin was lowered either with an OAqZO or an OAqPeracidO treatment. Kappa numbers between 3 and 5 were achieved without any viscosity penalty. The bleachability was better after ozone activation. A concentration of 0.25% ozone was more effective than 1.5% peracetic acid. Final brightness was above 90% ISO at similar strength properties. Final bleaching with hydrogen peroxide was made with only one stage. The addition of magnesium sulphate and sodium silicate improved the results. The lowest hydrogen peroxide demand resulted at temperatures below 100°C with a retention time of up to 24 h. The 90% ISO brightness level needed an input of less than 2% H_2O_2. Bleaching at 110°C accelerates the reaction, but yields inferior results in terms of peroxide consumption and viscosity.

RÉSUMÉ: Nous avons effectué des expériences de blanchiment avec de la pâte délignifiée à l'oxygène de façon conventionnelle. La quantité de lignine résiduelle a été réduite avec un traitement OAqZO ou encore avec un traitement OAqPeracideO. Des indices Kappa variant entre 3 et 5 ont été obtenus sans affecter la viscosité. L'aptitude de la pâte au blanchiment était améliorée par un stade d'activation à l'ozone. Une quantité de 0.25% d'ozone donnait de meilleurs résultats qu'une quantité de 1,5% d'acide peracétique. À des conditions de résistance similaires, le degré de blancheur final ISO était supérieur à 90%. Nous avons procédé au blanchiment final de la pâte au moyen d'un seul stade au peroxyde d'hydrogène. L'addition de sulfate de magnésium et de silicate de sodium a permis d'améliorer les résultats. La plus faible demande en peroxyde d'hydrogène a été observée à des températures inférieures à 100°C avec un temps de séjour atteignant les 24 heures. Pour obtenir un degré de blancheur d'un niveau ISO de 90%, nous avons utilisé moins de 2% de H_2O_2. Nous avons observé par ailleurs que le blanchiment à 110°C accélérait la réaction, mais donnait des résultats inférieurs en termes de consommation de peroxyde et de viscosité.

KEYWORDS: ALKALINE PULPS, CHEMICAL REACTIONS, CHEMICAL TREATMENT, CHLORINE FREE BLEACHING, DELIGNIFICATION, EUCALYPTUS, HYDROGEN PEROXIDE, KRAFT PULPS, MULTISTAGE PROCESS, OXYGEN, OXYGEN COMPOUNDS, OZONE, PEROXIDES, PEROXY ACIDS, PROCESS VARIABLES, PROCESSES, VARIABLES.