High-consistency ozone-bleaching technology

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ABSTRACT: Medium-consistency and high-consistency ozone stages are evaluated based on data from the literature. The effects of mass-transfer processes, ozone consumption, selectivity, and carryover of chemical oxygen demand are considered. Tests with the high-consistency (HC) ozone stage are aimed at investigating the optimum parameters. Data are presented for the rate of the reaction, the correlation between viscosity and strength properties for elemental-chlorine-free pulps and totally chlorine-free pulps, the influence of dynamic and static reactors, the effect of temperature, and the effect of closing water loops.

KEYWORDS: Chemical consumption, chemical oxygen demand, high consistency, mass transfer, medium consistency, ozone bleaching, reaction mechanism, selectivity, technology.

The demand for totally chlorine-free (TCF) pulps has increased in the past two years, and the supply of TCF pulps is increasing *(1)*. The current trend is mainly market driven, but it will be followed by legal regulations in many countries *(2)*.

Many mills have switched to an elemental-chlorine-free (ECF) bleaching process, but the leap to a TCF process will require a substitute for chlorine dioxide. Ozone is one bleaching chemical that can provide at least some of the delignification and brightening effects of chlorine dioxide.

The literature on ozone bleaching has yet to establish whether ozone bleaching is best carried out at high consistency or medium consistency. This article reviews data in the literature and considers the advantages of each approach. The discussion then moves on to examine the important parameters of a high-consistency (HC) ozone stage, with emphasis on the influence of

- Pulp consistencies greater than 30%
- Fluff quality
- Mixing during the reaction period
- Bleaching temperature.

The discussion of HC ozone stages is based on data from laboratory and pilot-scale trials. The article also addresses the effects of reaction speed and the closing of water loops.

All tons are metric; chemical consumption figures are based on moisture-free.

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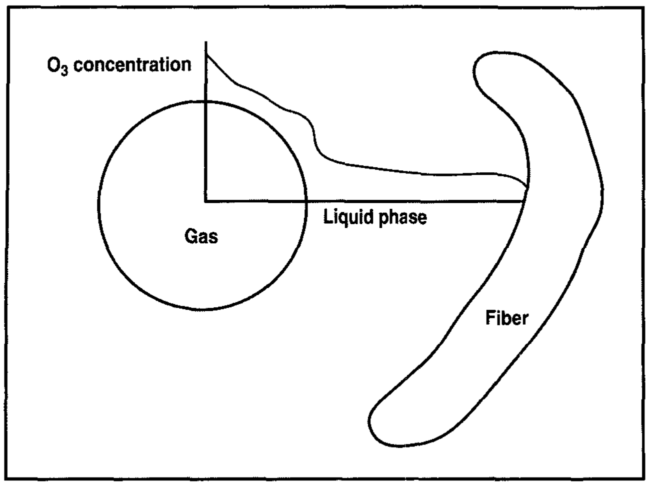
Comparison of medium- and high-consistency bleaching technology

Many papers dealing with medium- or high-consistency bleaching technology have been published during the last two years. Hardly any publication shows interest in the low-consistency technology, which can be attributed to the high demand of fresh water used for dissolving the ozone. Therefore, the low-consistency technology is not considered in this article.

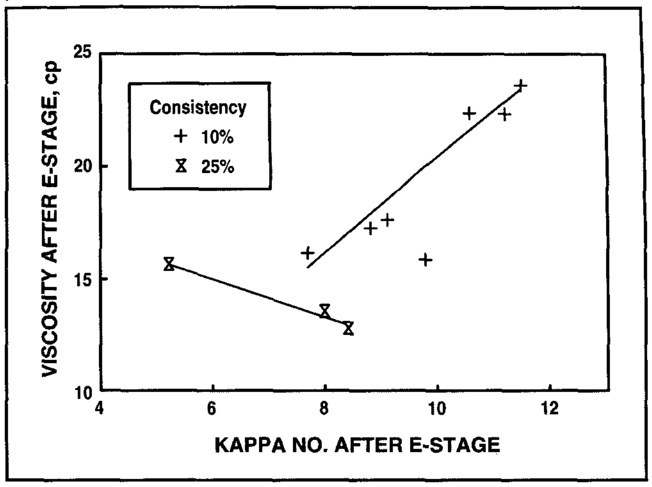
Mass transfer

Ozone is very reactive and can oxidize organic materials within a very short time if the reaction is not limited by mass-transfer processes. In medium-consistency bleaching, several mass-transfer steps are necessary preceding the reaction (**Fig. 1**). Outside of a high-intensity mixing zone, the ozone has to be transported by diffusion from the center of the gas bubble to the border layer. Be-

1. Mass transfer and ozone concentration profile for medium-consistency bleaching



2. Pulp viscosity (Scandinavian softwood, 0.8% ozone charge) as a function of kappa number at two levels of pulp consistency. [Data from Hurst *(14)*, who used various test procedures while holding test parameters constant.]



I. Ozone consumption for medium- and high-consistency trials

Literature	*Consistency*	*Testing conditions*	*Ozone consumed*	*Comment*
Oltmann *et al. (3)*	Medium	Laboratory; 5–30 s mixing in fluidized state	65%–90%	Increased consumption during mixing, improved delignification
Dillner *et al. (5)*	Medium	Laboratory; 5–20 s mixing in fluidized state	60%–99%	Increased temperature, raised consumption but lowered delignification
Gause *et al. (6)*	Medium	Laboratory; mixing with 97 kW·h/ton pulp	77%–88%	Increased consumption by lower gas/liquid ratio
Funk *et al. (7)*	Medium	Pilot plant; gas volume ratio 15%–45%	67%–99%	No data given of the effect of increased consumption on delignification
Peter, W. *(8)*	Medium	Mill experience	55%–75%	Approx. 10% losses during compression and piping
Helander *(9)*	Medium	Mill experience	90%–97%	No data given on ozone charge and delignification
Pikulin *et al. (10)*	High	Mill experience	90%–95%	Countercurrent flow of ozone and pulp; reactor with mixing
Kappel *et al. (11)*	High	Pilot plant	95%–99%	Co-current operation; mixing during fluffing, static reactor

cause of the very small size of the bubble, it cannot be assumed that this process is enhanced by convection. A drop in the ozone concentration inside the bubble yields the result shown in Fig. 1. The ozone concentration drops further during the mass transfer to the liquid phase.

Mixing in the liquid phase can be easily accomplished by some small turbulence, resulting in the moderate decrease of the concentration shown in Fig. 1. This holds true only if the pulp has been washed very well with fresh water. COD (chemical oxygen demand) in the liquor will consume ozone and cause a steeper decrease in concentration. The final step is the mass transfer to the fiber.

The complexity of these mass-transfer processes makes it necessary to use all means available to enhance them, such as:

- High turbulence (e.g., two mixers)
- Highest possible ozone concentration
- High gas pressure

A retention time in the tower without intensive mixing does not con-

II. Selectivity of medium- and high-consistency laboratory trials

Literature	*Measurement of*	*Findings*	*Comment*
Greenwood *et al. (13)*	Viscosity	HC lower viscosity	Procedure of HC test not specified, highly sophisticated procedure for medium consistency trials
Hurst, M. *(14)*	Viscosity	HC lower viscosities, but also lower kappa	Test procedure had biggest influence on kappa and viscosity
Oltmann *et al. (3)*	Viscosity, tear, tensile	HC slightly higher viscosity and tensile	Testing procedure optimized for both consistencies
Liebergott *et al. (15)*	Viscosity	Same viscosity at a given kappa number	Test procedure not described

III. Impact of COD source on the specific ozone consumption

Literature	*Tests*	*COD from*	*Impact of 10 kg/ton COD on specific ozone consumption*
Funk *et al. (7)*	Pilot	O-stage	+ 200%
Munro *(16)*	Pilot	O-stage	10 kg/ton COD: + 20% 18 kg/ton COD: + 300%
Gause *(6)*	Laboratory	O-stage	+ 47%
Gause *(6)*	Laboratory	Z-stage	+ 37%
Funk *et al. (7)*	Laboratory	Z-stage	0
Funk *et al. (7)*	Laboratory	E/P-stage	+ 30%
Sixta *et al. (17)*	Laboratory	EOP-stage	+ 100%

tribute to further delignification, as shown in the literature *(3, 4)*.

By implementing these enhancements, the medium-consistency stages can achieve acceptable results at low and moderate ozone charges. Since existing equipment can be used in many cases, investments in the fiber line are low, but the costs for the ozone generation and compression are higher.

High-consistency bleaching usually requires the installation of a complete new bleaching stage, but the mass transfer is very simple, and it is therefore easy to achieve high consumption figures. A chemical pulp fiber can store all water inside the fiber at a consistency of about 25%. It is therefore very unlikely that the fiber is surrounded by a thin filtrate layer at 40% consistency and at the surface tension of a liquor at the low pH of an ozone stage. Thus, high consistency requires only small turbulence to mix the ozone–oxygen gas mixture to avoid a concentration gradient and lead it to well-fluffed fibers.

Ozone consumption

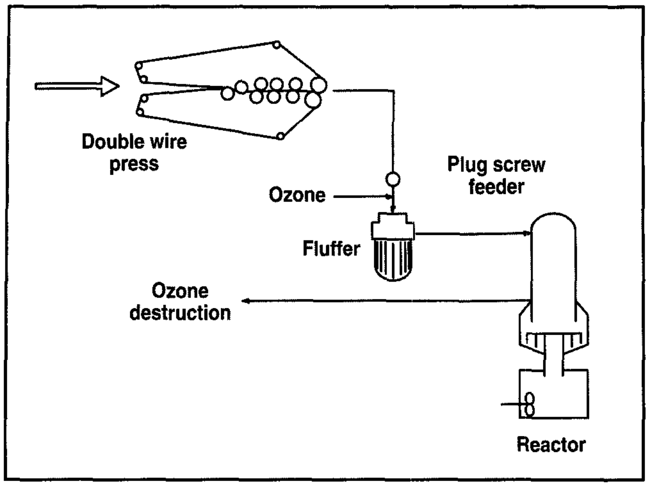
If the speed of the reaction is limited by mass-transfer steps, a considerable amount of ozone can leave the reactor without being utilized. Ozone also can be decomposed during compression in the reactor, or it can react with impurities in the pipes or in the pulp.

Table I summarizes data from the literature about ozone consumption at medium and high consistency. Two papers *(3, 5)* show that for a pulp at medium consistency, high ozone consumption requires a long mixing time in the laboratory (20–30 s). With these long mixing times, the consumption is in the range of 90–95%, and the kappa number decreases with mixing time. Increasing the temperature resulted in higher consumption figures but had an adverse effect on delignification *(5)*.

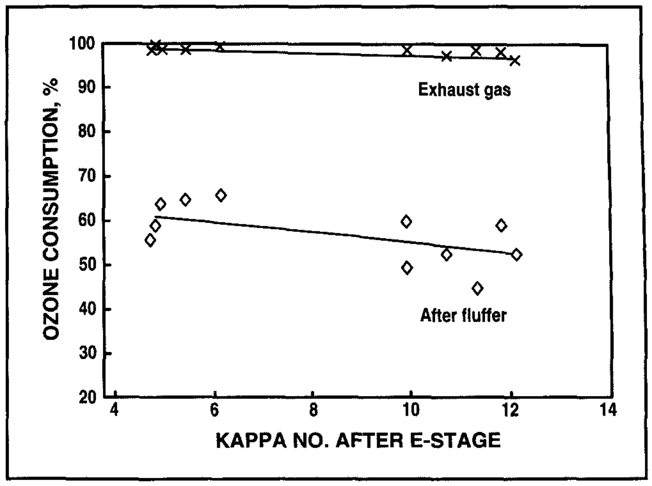
Laboratory trials by Gause *et al. (6)* showed an influence of the gas-to-liquid ratio on the ozone consumption. Mixing energy was very high for these trials (approximately 100 kW·h/ton pulp).

In contradiction to these results, Funk *et al.* reported high ozone consumption for medium-consistency pilot trials *(7)*, which were obtained

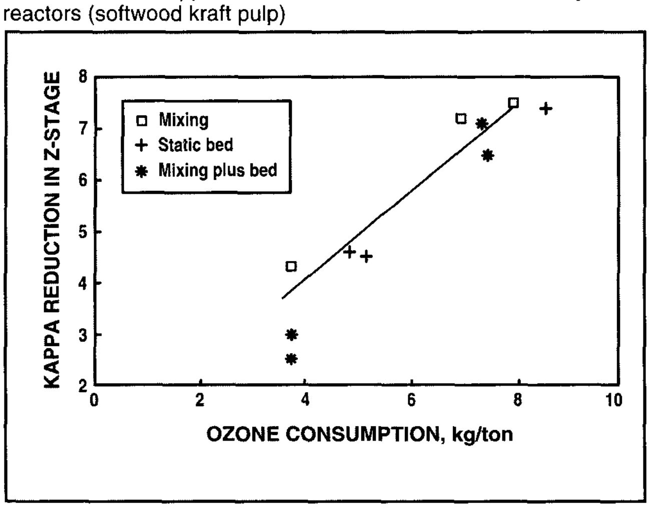
3. Flowsheet of the pilot ozone stage



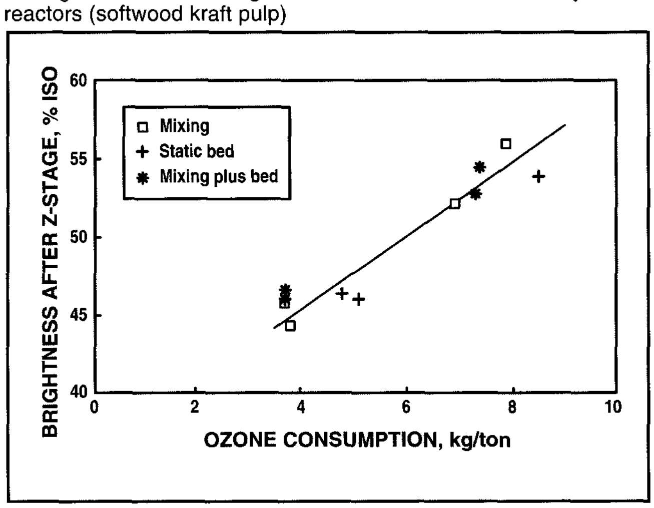
4. Ozone consumed in the fluffing zone and in the static moving bed reactor (softwood kraft pulp)



5. Reduction in kappa number for simulation of static and dynamic reactors (softwood kraft pulp)



6. Brightness after Z stage for simulation of static and dynamic reactors (softwood kraft pulp)



by increasing the ozone concentration and decreasing the gas volume. No data are given to show if these higher consumption figures also resulted in better delignification. One set of test data from the same pilot plant *(12)* shows an ozone consumption of only 68%. Losses during compression and piping are not included in these consumption figures.

Data from the 300-ton/day medium-consistency plant at Lenzing, Austria, *(8)* showed approximately a 10% loss of ozone during compression and ducting of the compressed gas. Between 15–35% of the ozone leaves the reactor again. Ozone consumption for the reaction itself is therefore limited to 55–75%.

Data from another mill installation *(9)* show very high consumption figures, but no data are given on whether this also results in better delignification.

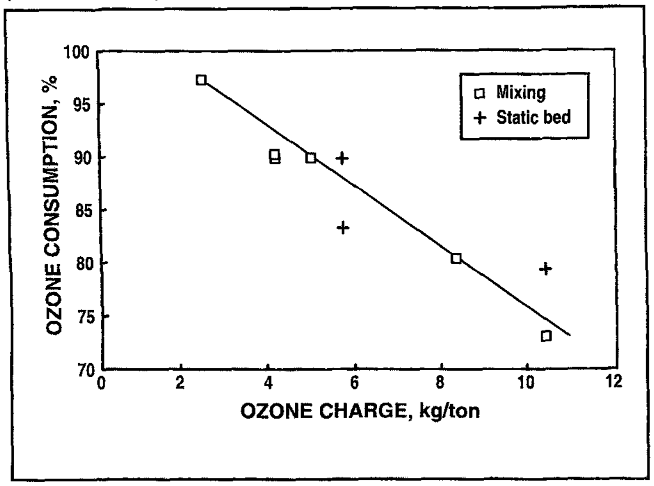
The high-consistency bleach plants report ozone consumptions of 90–95% at Union Camp's Franklin Mill *(10)* and 95–99% at the pilot plant of ÖZF, Austria *(11)*. Besides the big difference in the size of these plants, the concept of reactor design differs considerably. In the Union Camp plant, the ozone gas is led countercurrent to the pulp and mixed in a reactor. In the pilot plant at ÖZF, the ozone is already added to the fluffer ahead of the concurrent static moving bed reactor.

Selectivity

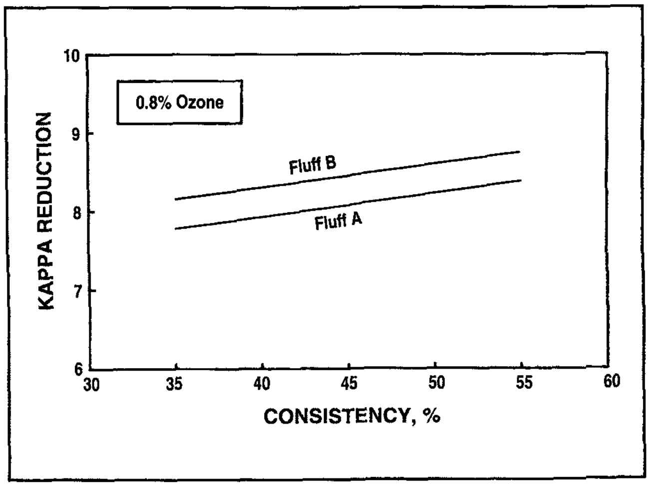
Most of the works listed in **Table II** evaluated the selectivity of the ozone reaction by measuring viscosity. However, the relationship between viscosity and strength properties is changed completely in TCF bleaching sequences *(15)*.

The test procedure has a decisive influence on both the delignification and the selectivity, as shown in **Fig. 2.** The data for this figure were taken from Hurst *(14)*. All these tests used the same pulp sample and the same parameters (ozone charge, pH, tem-

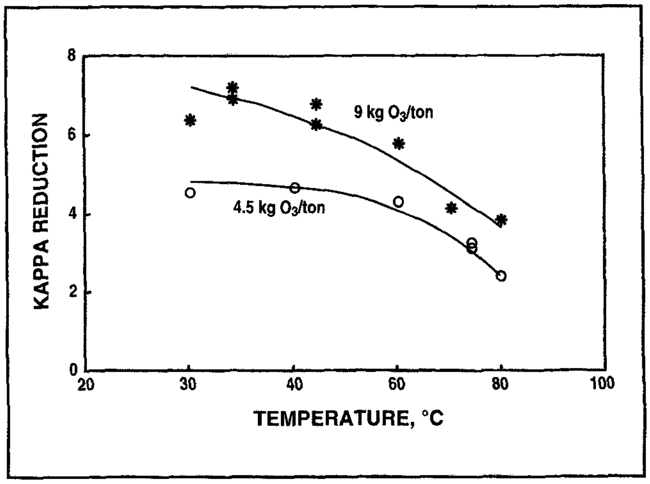
7. Ozone consumption for simulation of static and dynamic reactors (softwood kraft pulp)



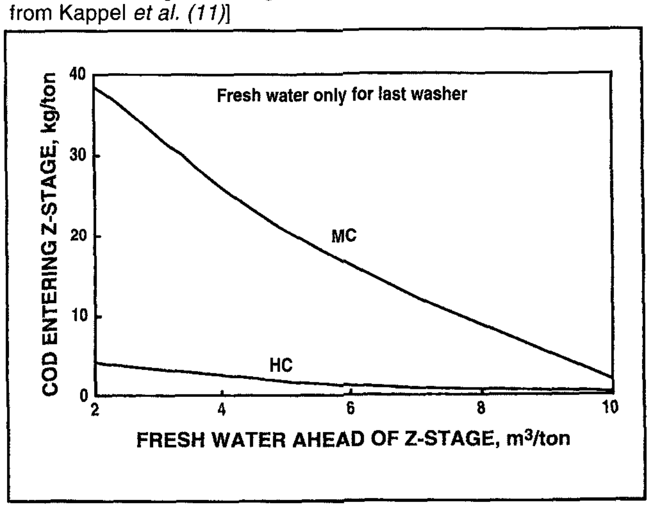
8. Influence of fluff quality and consistency on delignification (softwood kraft pulp)



9. Influence of temperature on delignification (softwood kraft pulp)



10. COD entering the Z-stage for an effluent-free bleach plant [data from Kappel *et al.* *(11)*]



perature). Only the test procedure was varied, mainly the mode of applying ozone (number of pulses). Despite Hurst's conclusion that medium consistency would be more selective, the following is obvious from the data shown in Fig. 2:

- The test procedure is the key to both delignification and selectivity.
- The HC trials reached the lowest kappa numbers.
- Under optimum test conditions, viscosity is even higher at 25% consistency for a given kappa number.

The impact of the test procedure may explain the lower viscosities obtained with HC trials in the work of Greenwood and Szopinski *(13)*. In those trials, substantial efforts were made to ensure very accurate dosage of ozone and high-intensity mixing over a reaction time of up to 60 s. On the other hand, the fluff quality, which is a key parameter to achieve good selectivity, is not specified in that paper.

Oltmann *et al.* *(3)* optimized bleaching conditions for both consistencies. They found slightly higher viscosities and tensile values for the HC trials. The tear index is influenced not only by the bleaching reaction but also by the strong agitation at medium consistency.

Influence of impurities

COD carryover to the Z stage increases the specific ozone consumption required to delignify one kappa unit. Both the origin and the amount of COD influence this increase in ozone consumption. There are very contradictory data published in the literature, as seen in **Table III**. COD carried over from the oxygen stage showed a detrimental effect, with COD carryover of 10 kg/ton tripling the specific ozone consumption in a pilot plant *(7)*. A later work using data from the same pilot plant *(16)* showed only a 20% increase in ozone consumption at COD carryover of 10 kg/ton but a 300% increase at carryover of 18 kg/ton and almost no delignification effect at carryover greater than 20 kg/ton.

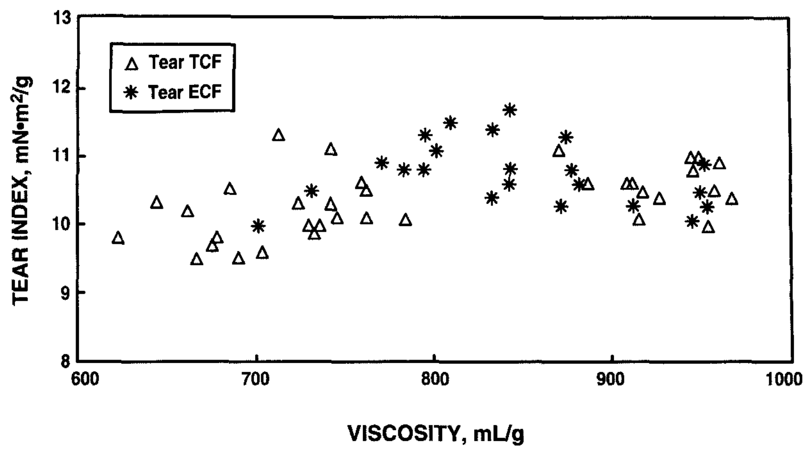
Gause *et al.* *(6)* found a 47% increase for 10 kg COD carryover from the O stage and a 37% increase for carryover from the Z stage.

COD from an EP stage *(7)* or an EOP stage *(17)* shows less but still significant influence in the laboratory.

COD that had already been in a Z-stage once is reported to have no effect *(7)*. This is in contradiction to the findings of Gause *et al.* *(6)*. Since ozone has the second strongest oxidation potential *(15)*, it is doubtful if these laboratory results can also be achieved in pilot- or mill-scale installations.

From this great variety of data, it might be concluded that COD carryover of 10 kg/ton may increase the cost of this bleaching stage by 30–100%.

11. Tear index as a function of viscosity for TCF and ECF bleaching (softwood kraft pulp)



Experimental

The results presented in this paper were measured in the laboratory and in a pilot plant. In laboratory tests, pulp samples were treated in an acid stage, dewatered to the desired bleaching consistency (about 40% in most cases), fluffed thoroughly, and mixed with the oxygen–ozone gas mixture in a temperature-controlled, rotating glass flask. Residual ozone was absorbed in an aqueous solution of potassium iodide and titrated. For simulation of the static bed reactor, a 100-mm tube was filled with the fluffed pulp. The tube was equipped with a screen at the bottom to support the pulp and allow distribution of the ozone. Ozone entered the tube for half of the reaction time at the top and half at the bottom.

The pilot trials were run at ÖZF, a seven-stage bleach plant with a production of 15 tons/day. It is operated continuously for at least four days to simulate the filtrate loops of a pulp mill. The pilot ozone stage, illustrated in **Fig. 3**, consists of a double-wire press to dewater the pulp suspension after acidification. After sealing with a plug-screw feeder, the pulp is fluffed and simultaneously mixed with the ozone. The reactor is a static moving bed with a retention time of several minutes. The gas is separated and led to ozone destruction. The pulp is discharged and diluted in a chest.

All the tests used an oxygen-delignified softwood kraft pulp sample.

Discussion

Bleaching reaction

A series of tests were aimed at acquiring information on the rate of reaction and the optimum reactor design. As described in the Experimental section, ozone is mixed with pulp in the fluffer. This allows a major part of the reaction to take place within a very short period of time where, due to lack of storage volume, almost ideal plug flow can be assumed. As seen in **Fig. 4**, 50–65% of the ozone is already consumed in the fluffing zone. In the subsequent static moving bed, the residual ozone can react with the pulp. The residual ozone after the bed is only 1–2%. The ozone in the static moving bed is actually used for delignification, since approximately 40% of the total reduction in kappa takes place there, as shown by Kappel *et al.* *(11)*.

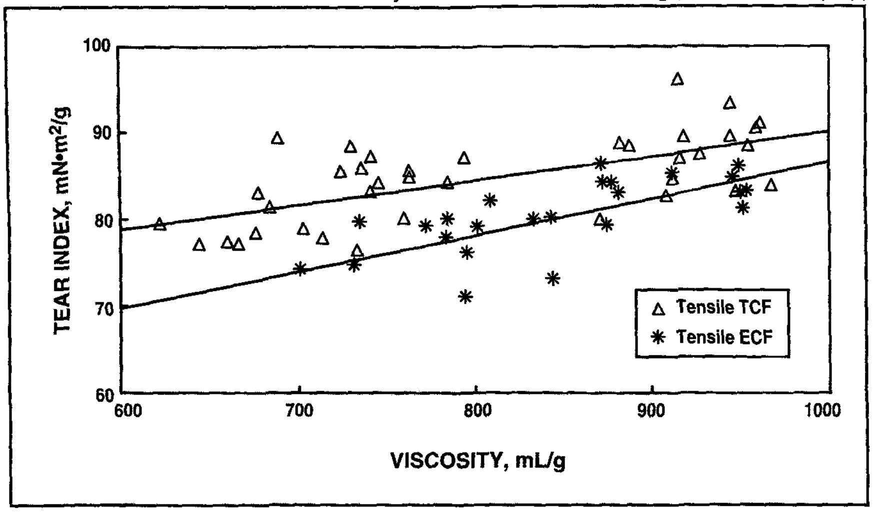
It was not possible to compare a dynamic reactor with the static reactor in the pilot plant, since an accurate comparison requires that both systems be optimized. Instead, both reactors were simulated in the laboratory. The dynamic reactor was simulated by a rotating glass vessel, which has somewhat lower turbulence but has a more constant retention time than the mill-scale unit.

A vertical tube with a filling height of 200 mm was used to simulate the static reactor. The velocity of the gas penetrating the fluffed pulp was chosen in the same range as in mill scale. The limited height did not allow us to achieve very high consumption figures in the laboratory.

Figure 5 shows the reduction in kappa number for mixing during the reaction, in the static bed, and for a combination of mixing (60% ozone) followed by a static bed (40% ozone). Besides the scattering of test data, all three methods showed equivalent results. The average kappa number in the static bed was slightly higher because the pulp in the middle of the column was never exposed to fresh ozone.

All reactors also showed similar results with regard to brightness, as seen in **Fig. 6**. Because of the conditions in the laboratory (limited height of the static bed), ozone consump-

12. Tensile index as a function of viscosity for TCF and ECF bleaching (softwood kraft pulp)



IV. Comparison between medium- and high-consistency ozone bleaching

	Medium consistency	*High consistency*
Reaction mechanism	Limited by complex mass transfer	Simple mass transfer
Reaction conditions	High turbulence high gas pressure high O_3 concentration	Good fluffing plug flow
Selectivity	Equal to HC	Equal to MC
Ozone consumption	50%–97%	90%–99%
Closing of water loops	High amount of COD	Still low COD

tion was lower than in the pilot plant, but no difference could be found between static and dynamic conditions, as seen in **Fig. 7**.

The selectivity was found to be high for the high-consistency trials. It is essential for good bleaching results and high selectivity to have excellent fluff quality and high consistency. In the laboratory, two different fluff qualities were produced for various high pulp consistencies. There is no standardized method for measuring fluff quality, but both of the pulps used in the trials can be described as well-fluffed pulp, with Fluff B showing superior disintegration.

Experimental design was used in the planning of these tests, and the results were incorporated into a second-order equation. The correlation coefficient was very high (>0.98), proving that the effects described by the equations are truly significant.

Figure 8 shows the influence of bleaching consistency and fluff quality (Fluff B > Fluff A) on the reduction of kappa number. The higher the consistency, the better was the delignification, even when using the same fluffing method. Since the pulp had been washed very well before bleaching, the slopes of the curves in Fig. 8 are due to the consistency during the reaction and, possibly, to differences in fluff quality. The slopes cannot be attributed to the amount of impurities present during the reaction, which would further increase the consistency effect. Within the consistency range of 35–55%, the effect of consistency was 0.6 kappa number points.

The superior quality of Fluff B increased the kappa-number reduction by 0.35 points.

Bleaching temperature

The effect of bleaching temperature was measured in the laboratory and in the pilot plant for two different levels of ozone addition. As published in the literature *(e.g., 20)*, lower temperatures produce better delignification (**Fig. 9**). At the lower ozone charge of 4.5 kg/ton, delignification deteriorated significantly at temperatures above 50°C, whereas a higher ozone charge of 9 kg/ton allowed maximum temperatures of 40°C. At temperatures near 60°C, doubling the ozone charge produced almost no beneficial effect.

Fig. 9 shows pilot and laboratory tests. Although very well-washed pulp was used in the laboratory, the results are well in line with pilot-plant operation.

Closing of water loops

A TCF bleaching sequence offers the possibility of closing the water loops and running more filtrates countercurrent to the evaporation plant. After the elimination of chlorine compounds in the bleaching sequence, this will be the next step for environmental protection. Besides the problems with the sulfur and sodium balance *(18)* and the buildup of nonprocess elements *(19)*, there will be significantly increased amounts of COD recirculated ahead of the ozone stage. The results in Table III suggest that ozone consumption of COD might exceed that of delignification.

If fresh water is applied only at the last washer after the final P stage, the filtrate used for washing ahead of the Z stage is contaminated with COD dissolved in the Z, EO, and P stages. With a minimum of 2 m^3/ton fresh water addition ahead of the Z stage, the carryover into the Z stage can accumulate up to 38 kg/ton in medium-consistency bleaching, as seen in **Fig. 10**. This is almost four times higher than the range investigated in the literature (Table III). For medium consistency, this carryover can be reduced to less than 10 kg/ton if more than 8 m^3/ton fresh water is added ahead of the Z stage, which is in contradiction to the effluent-free bleach plant concept. The basis of the calculations for Fig. 10 can be found in the work of Kappel *et al. (11)*.

In high-consistency bleaching, most of the contaminated filtrate is removed by dewatering ahead of the Z stage. Even if little fresh water is added, the COD level is below 5 kg/ton. As described previously, this residual COD is mainly located inside the fiber and thus has only a very limited effect on the bleaching reaction, as seen in Fig. 9, where laboratory and pilot plant trials gave equivalent results.

Closing the water loop did not decrease the efficiency of the bleaching stage. The level of COD into the Z stage was usually 5–15 kg/ton. The high level of COD into the Z stage of the pilot plant can be explained by the fact that O-bleached pulp was repulped using filtrate, and there was only a very small purge stream ahead of the Z stage.

Strength properties

The strength properties were hardly changed in the high-consistency ozone stage, as shown in the work of Kappel *et al. (11)*. It was confirmed in various test runs that measurement of viscosity is not the right tool to evaluate what is happening with the pulp in the bleaching stages. **Figures 11 and 12** compare the relationship between viscosity and pulp strength (tear index and tensile index, respectively) for a four-stage TCF $[OZ_{HC}(EOP)P_{HC}]$ bleach sequence and a five-stage ECF $(OD_0ED_1D_2)$ bleach sequence. Tear index dropped only slightly when the viscosity was reduced by 300 cm^3/g. The ECF pulp showed much higher viscosities but only slightly higher tear index. The tensile index was slightly higher after the TCF bleaching sequence, although the viscosity was much lower. In TCF bleaching, the viscosity can be 150–200 cm^3/g lower at the same tensile index.

Conclusions

According to data from the literature, medium- and high-consistency ozone bleaching can be compared as shown in **Table IV**.

From the results of high-consistency bleaching trials in the laboratory and pilot plant, the following conclusions can be drawn:

- About 60% of the ozone is used in the high-turbulence zone during fluffing.
- The residual 40% is consumed for delignification in the static moving bed.
- Mixing during the reaction does not improve bleaching results.
- High consistency (>40%) and good fluffing are essential for good delignification and high selectivity.
- Closing the water loop had no effect on the ozone stage.
- Despite lower viscosities, strength properties were hardly changed in the Z stage. **TJ**

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