IPBC 2014

Bleaching Practices For Oxygen-Delignified Softwood Kraft Pulp: Analysis of the 2013 PAPTAC Bleaching Committee Survey

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The Bleaching Committee of the Pulp and Paper Technical Association of Canada (PAPTAC) conducts regular surveys of member-mill bleached kraft pulp operations. Since the last survey in 2003, the following changes have been observed for oxygen-delignified softwood kraft pulp:

- Sodium hydroxide use in the bleach plant has decreased by an average of 6.4 kg/ADMT or 26%;
- Chlorine dioxide use has decreased by an average of 0.5 kg/ADMT;
- Hydrogen peroxide use has decreased by an average of 0.9 kg/ADMT;
- Digester kappa no. has decreased by an average of 1.3 kappa units, and bleach feed kappa no. has decreased by an average of 1.2 kappa units;
- Pulp production has increased by an average of 6.5%;
- Final bleach plant brightness has increased by an average of 0.5% ISO.

Comparison of mills with low and high relative chemical consumption indicates that "low chemical consuming" oxygen-delignified softwood bleach plants:

- Feed the bleach with a pulp having a kappa no. 2.8 units higher than the "high relative consumption" bleach plants;
- Produce pulp with 0.5 points higher final brightness;
- Use 3.3 kg/ADMT less chlorine dioxide, but 1.7 kg/ADMT more hydrogen peroxide;
- Operate with a significantly lower D_0-stage Kappa Factor;
- Are more likely to control the D_0 stage using an online kappa analyzer in combination with optical and residual sensors;
- Are more likely to control the D1 stage using a combination of post-Eop kappa no. and post-Eop brightness, with feedback from the post-D1 brightness;
- Have a lower ratio of D1-stage chlorine dioxide to the Eop kappa number;
- Operate with a higher pH in the D2 stage.

Keywords: bleaching; best practices; optimization; chlorine dioxide; hydrogen peroxide; sodium hydroxide; oxygen delignification

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INTRODUCTION

In an effort to determine current "best practices" for kraft pulp bleaching in North America, the Bleaching Committee of the Pulp and Paper Technical Association of Canada (PAPTAC) conducts regular surveys of member-mill bleached kraft pulp operations. Previous surveys were carried out in 1995, 1996, 1999, and 2003 [1-5].

As in the past, a detailed questionnaire was prepared requesting information on the unbleached pulp, equipment, chemical consumption, and process conditions in each bleaching stage (including oxygen delignification), as well as washing, process control, and water and energy use. The survey was sent to 18 Bleaching Committee member mills producing fully-bleached oxygen-delignified softwood kraft pulp in Canada and the northern United States. Responses were received from 13 mills, representing 14 bleach plants. 11 of the 13 mills produce only market pulp, while the remaining two mills are at least partially integrated, producing tissue and market pulp and tissue and bleached board.

As shown in Figure 1a, all formats of oxygen delignification were represented; single- and 2-stage medium consistency (six and four bleach plants respectively), high consistency (two bleach plants), and low-pressure "mini-O_2" (two bleach plants). Figure 1b shows that half of the respondents operate 5-stage bleach plants – a reflection of the Canadian market pulp influence on the survey. Two mills operate 3-stage bleach plants – one using xylanase enzymes – and the remainder operate 4-stage plants, using both "DD" and "DnD" brightening sequences.

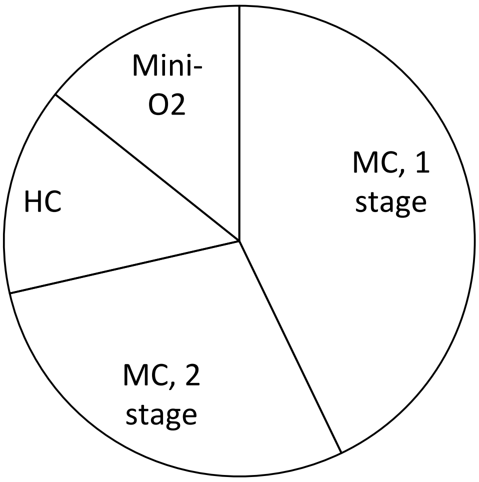

Figure 1a: Oxygen Delignification Systems

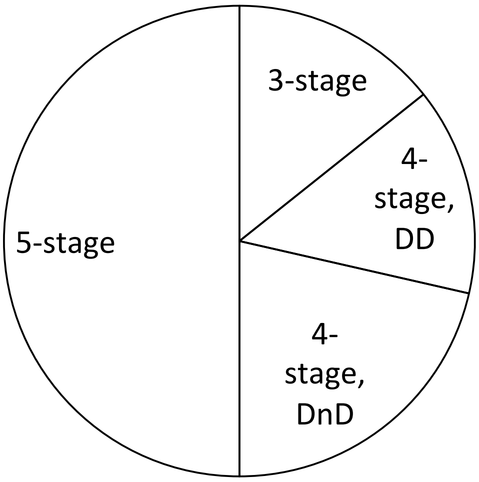

Figure 1b: Bleach Plant Configurations

PART I: SURVEY RESULTS AND TRENDS SINCE 2003

A decade has passed since the last PAPTAC bleaching survey was carried out. No new pulp mills have been built, and none of the mills that participated in this survey have done any major fibreline upgrades. So a comparison of the averages for the 2003 and 2013 surveys shows that many parameters have not significantly changed – with one very notable exception.

Digester kappa no. has decreased by an average of 1.3 kappa no. units, and the decrease has been relatively consistent across the entire group, as shown in Figure 2. This was somewhat surprising, given the focus on improving the yield of pulp from wood at many mills, but may reflect an increased focus on energy production at many market pulp mills. As well, comparing individual mill responses from the two surveys, the average pulp production rate has increased by 6.5%.

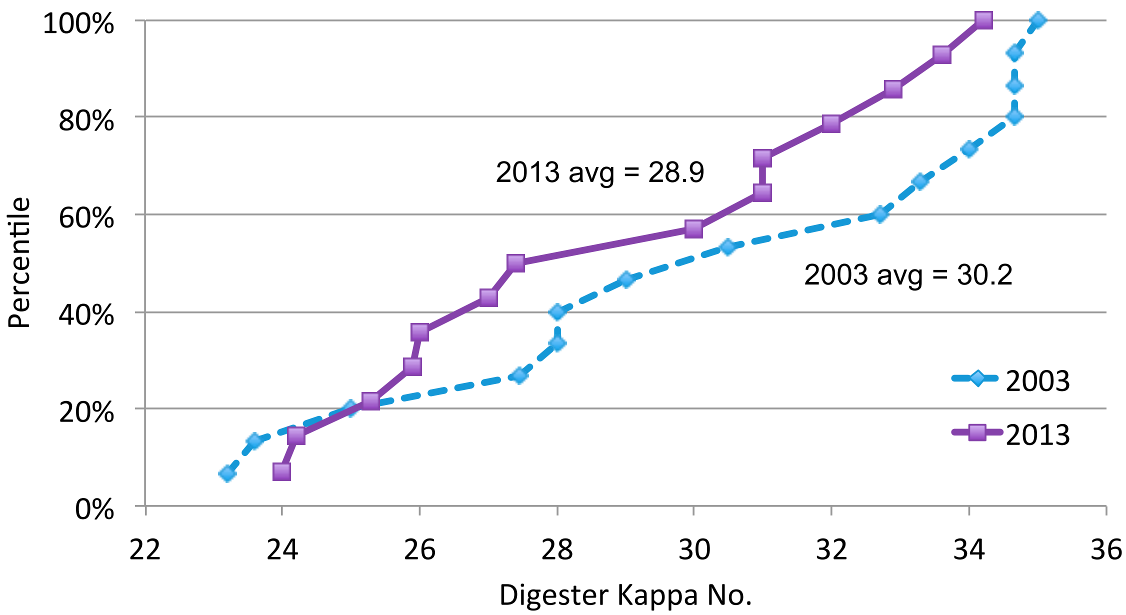

Figure 2 – Oxygen-Delignified Softwood, Digester Kappa Number

The extent of oxygen delignification has increased slightly, from 43% to 46% kappa no. reduction. Although this change appears to be fairly consistent across the group, as illustrated in Figure 3, it is in fact primarily due to just one mill, which upgraded their oxygen delignification system from one reactor to two to increase oxygen delignification efficiency, and so shifted the distribution. The average for the other mills that responded to both surveys has not changed.

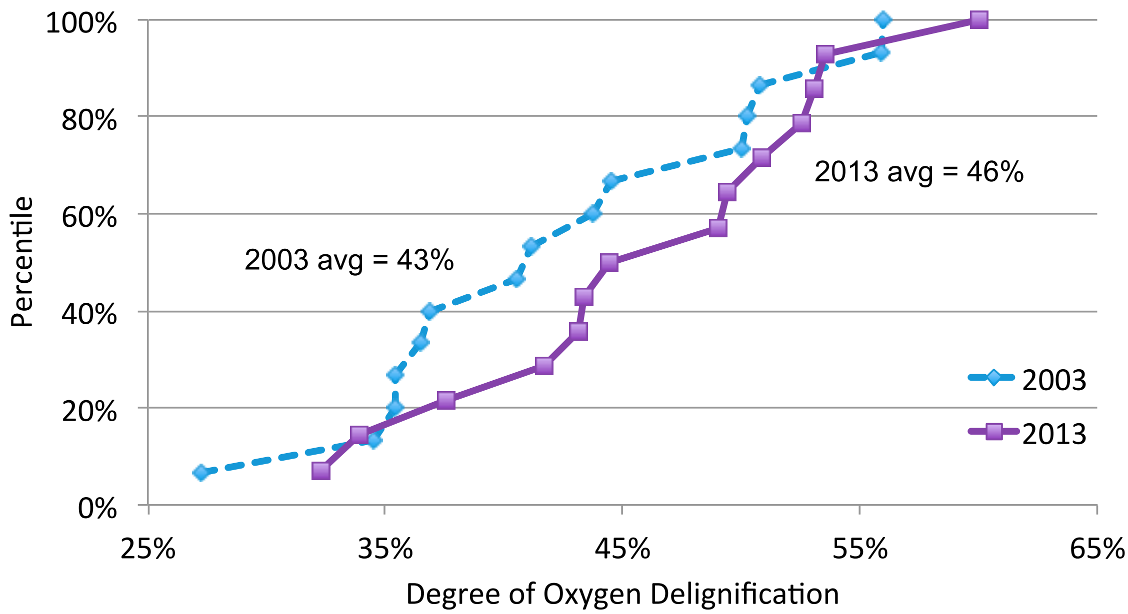

Figure 3 – Oxygen-Delignified Softwood, Oxygen Delignification Efficiency

As a result of the lower digester kappa no. and the higher average degree of oxygen delignification, the bleach feed kappa no. has decreased by an average of 1.2 kappa no. units (Figure 4) – a trend which has continued since 1999.

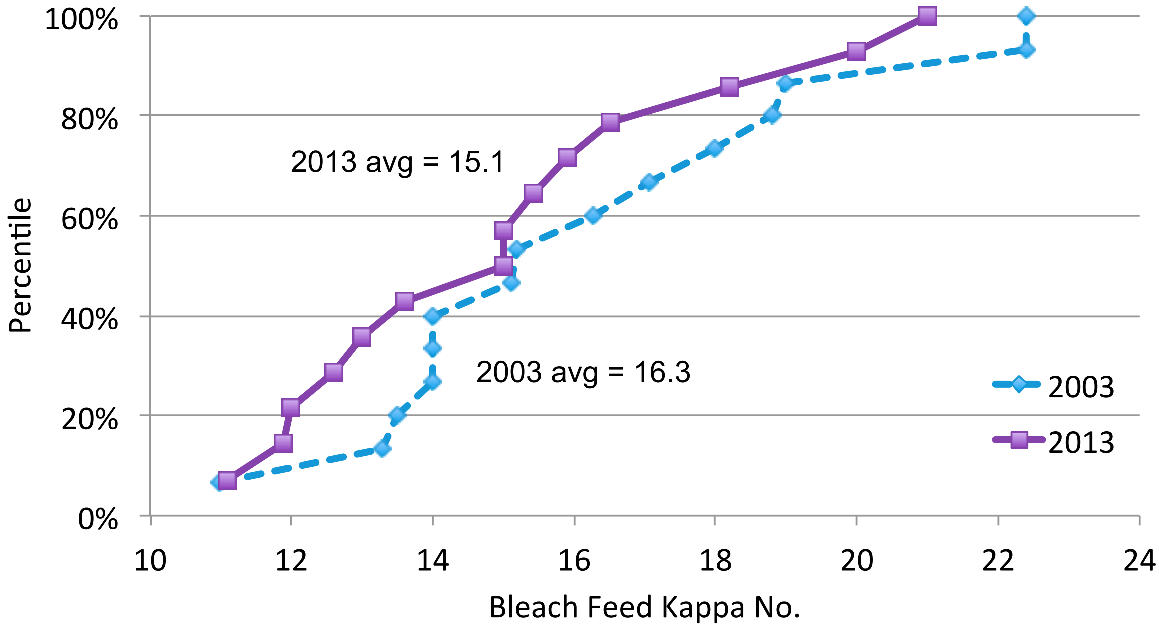

Figure 4 – Oxygen-Delignified Softwood, Bleach Feed Kappa No.

However despite the lower bleach feed kappa no., total chlorine dioxide use has only decreased by an average of 0.5 kg/ADMT[1] (Figure 5), although peroxide use has also decreased by an average of 0.9 kg/ADMT (Figure 6). The net result is that the average Sequence Kappa Factor[2] is essentially unchanged, although there are some changes in the distribution, as can be seen in Figure 7. In particular, the relative position of the third- and fourth-quartile mills has shifted (keeping in mind that the same mills are not necessarily included in both surveys). It appears that while some of the fourth-quartile mills have made improvements in bleaching chemical use, the third-quartile mills have slipped back and are using more chlorine dioxide.

There were two other changes in the data which help to explain these observations. First, the average final bleach plant brightness has increased by 0.5 points, from 88.6% ISO to 89.1%. At high brightness, this can noticeably impact bleaching chemical demand. Second, and perhaps more important, the average production rate – comparing mills which responded to both surveys – has increased by 6.5%. As discussed previously, there have not been any significant fibreline upgrades in this time, so this higher production rate translates to shorter retention times in bleach plant towers, and an increased loading on washers, both of which contribute to increased bleaching chemical demand.

1. All chemical charges are expressed as kg per air-dry metric tonnes (ADMT) of finished (bleached) pulp. Kappa Factors are also calculated on this basis.
2. Sequence Kappa Factor is a measure of oxidizing chemical use per unit kappa, and is defined here as total ClO_2 + H_2O_2 applied on an air-dry bleached pulp basis, expressed as % equivalent chlorine, and divided by the bleach feed kappa number.

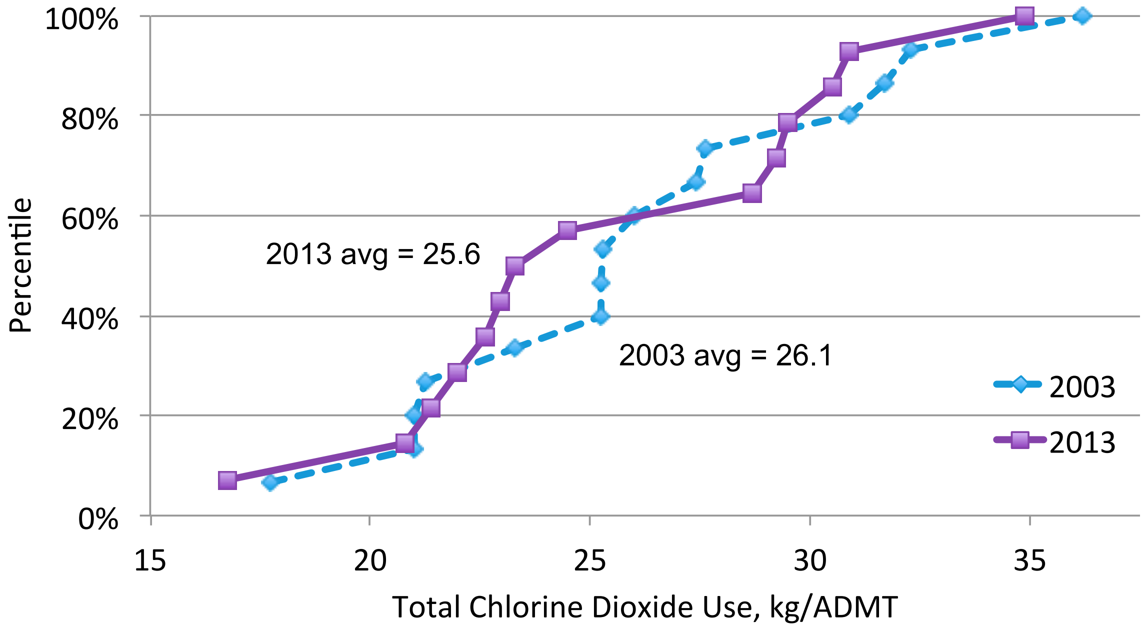

Figure 5 – Oxygen-Delignified Softwood, Total Chlorine Dioxide Use

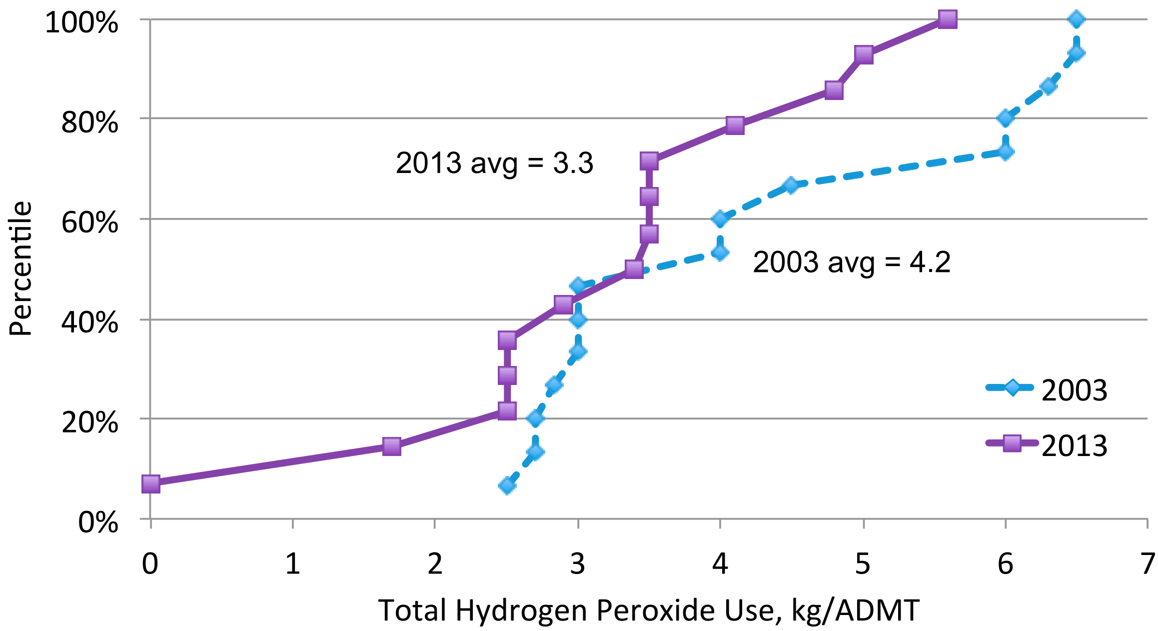

Figure 6 – Oxygen-Delignified Softwood, Total Hydrogen Peroxide Use

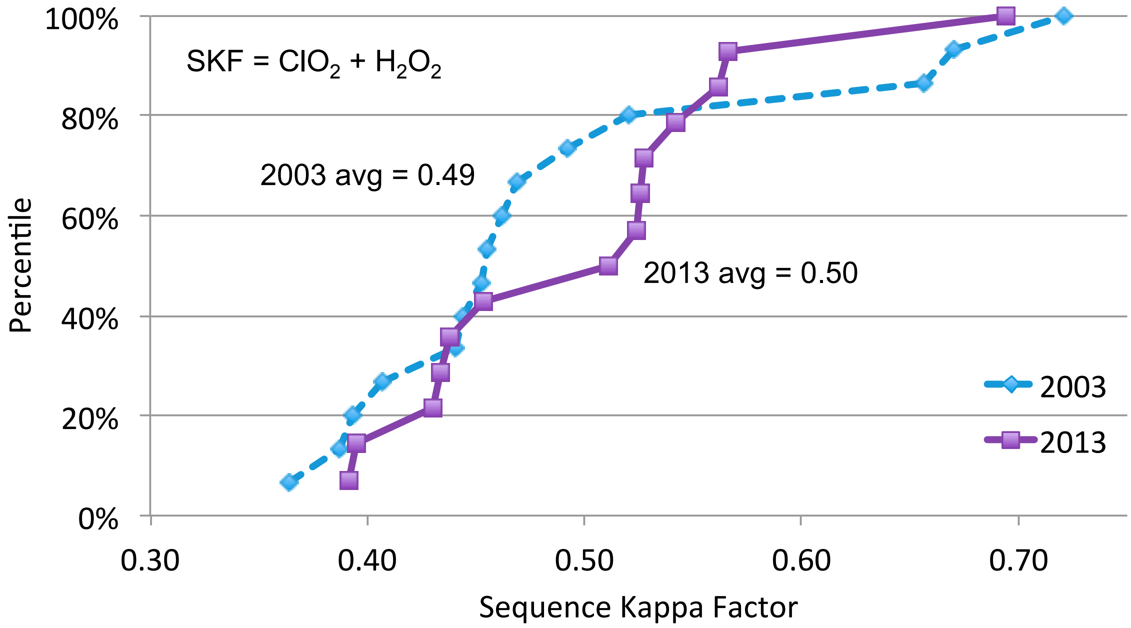

Figure 7 – Oxygen-Delignified Softwood, Sequence Kappa Factor
(Total ClO_2 + H_2O_2 per unit bleach feed Kappa No.)

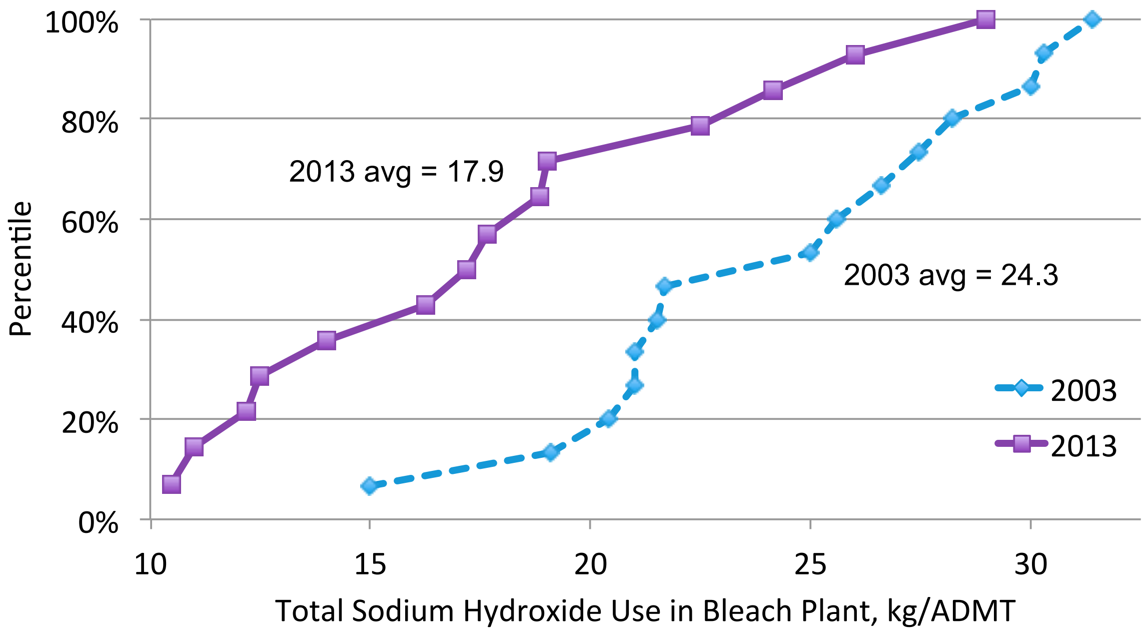

Figure 8 – Oxygen-Delignified Softwood, Sodium Hydroxide Use (bleach plant only)

The most significant change in chemical use since 2003 is a dramatic decrease in the amount of sodium hydroxide used in the bleach plant, as shown in Figure 8. On average, caustic use has decreased by 26%, and the decrease is again consistent across the range of respondents. As the D_0-stage chlorine dioxide use has not decreased commensurately, this means that the ratio of Eop-stage NaOH to D_0-stage ClO_2 has also dropped significantly, from 1.24 in the 2003 survey to 0.92 in the present survey. This is likely attributable to two main factors – the spike in sodium hydroxide prices in the mid-2000's, which caused many mills to examine their caustic usage more carefully, and also an improved understanding of the impact of sample temperature on pH measurement [6], which has led many mills to adjust the target terminal extraction-stage pH and optimize the dosage of sodium hydroxide.

PART 2: OXYGEN DELIGNIFICATION AND BLEACH PLANT CHEMICAL USE

Oxygen delignification efficiency (kappa no. decrease) ranged from slightly more than 30% (for the two "mini-O2" systems) to a high of 60%, as shown in Figure 9. As might be expected, this resulted in a wide range of chlorine dioxide usage, from a low of 17.6 kg/ADMT to a high of 35.0 kg/ADMT (Figure 10). However, the mills with a high degree of oxygen delignification – which typically suggests a lower kappa no. entering the bleach plant – were not necessarily the mills with low bleaching chemical use. As shown in Figure 11, there is a definite downward trend in the relationship between total chlorine dioxide use and bleach feed kappa no. – chlorine dioxide use is decreased by approximately 1 kg/ADMT for each unit decrease in kappa no. – but the relationship is not very strong; some mills bleaching 11-15 kappa pulp are using as much chlorine dioxide as mills bleaching 20 kappa pulp. This will be explored further in the next section.

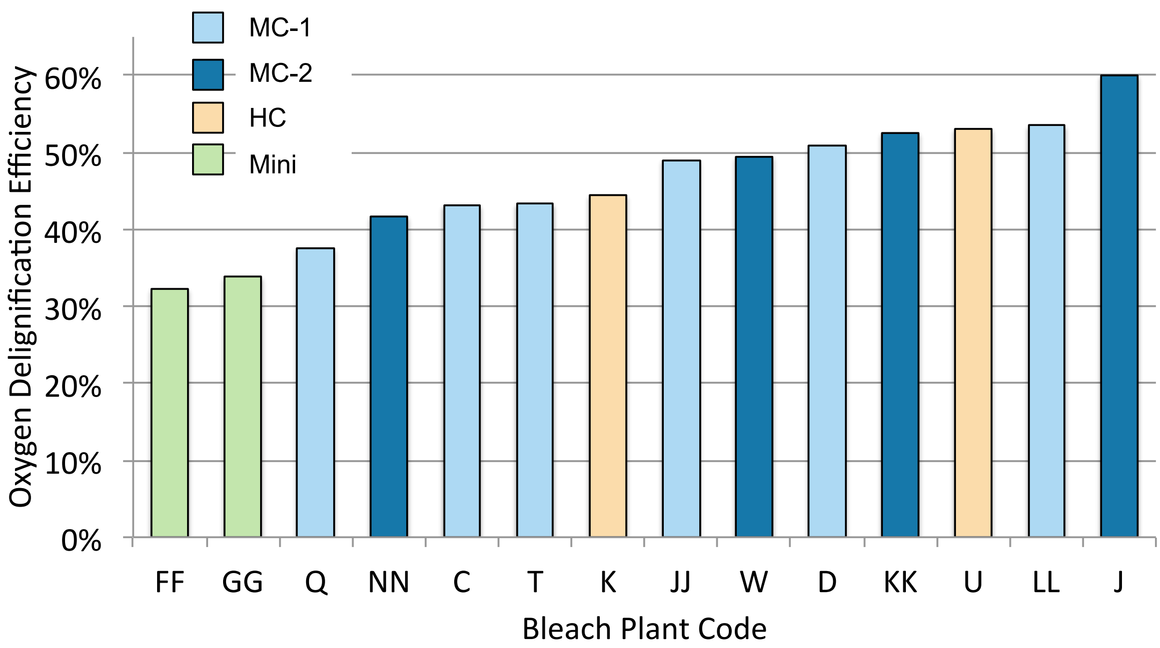

Figure 9: Oxygen Delignification Efficiency, Softwood Kraft Pulps

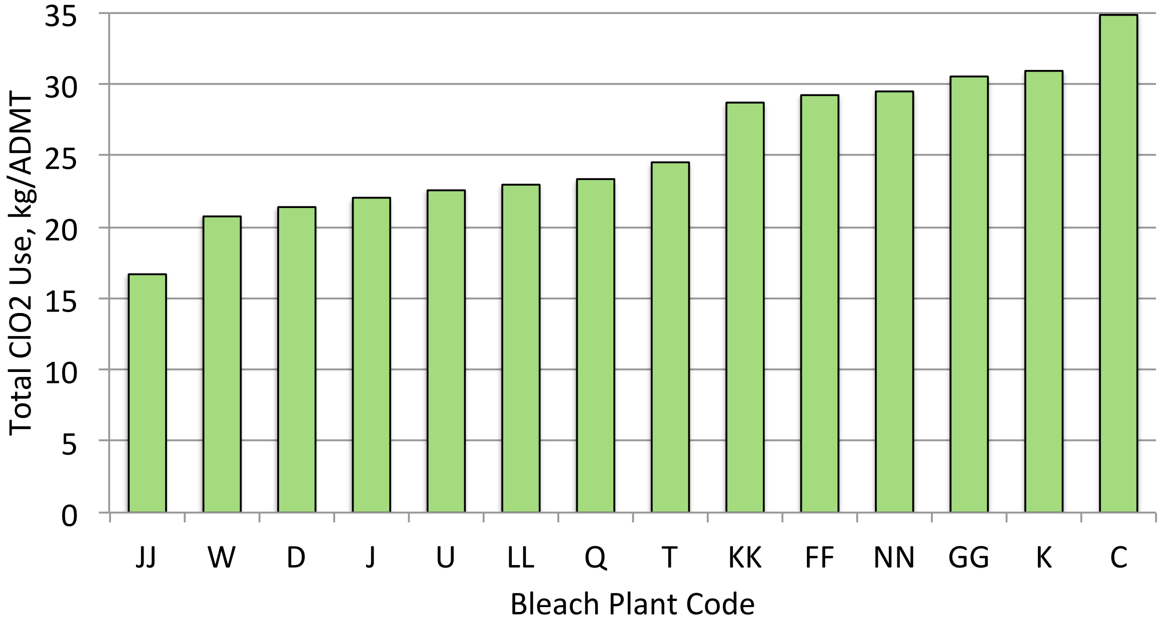

Figure 10: Chlorine Dioxide Use for Oxygen-Delignified Softwood Kraft Pulps

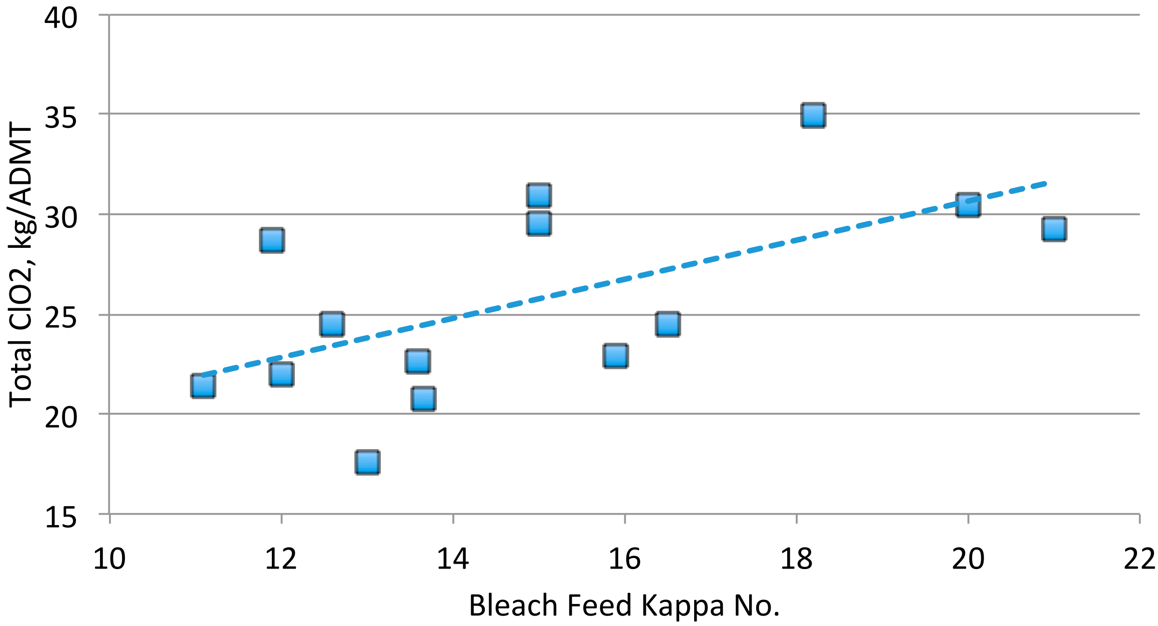

Figure 11: Chlorine Dioxide Use for Oxygen-Delignified Softwood Kraft Pulps

The relationship between kappa number and bleaching chemical use appears different when expressed as Sequence Kappa Factor. Figure 12 shows that bleaching chemical use per unit kappa increases as the kappa no. of the pulp entering the bleach plant decreases; in general, pulps appear to become more difficult to bleach as the kappa no. is decreased. This is not a surprising observation; it has long been known that due to the nature of the reactions between oxygen and lignin and chlorine dioxide and lignin, oxygen-delignified pulps require more chlorine dioxide per unit of lignin remaining in the pulp, compared to "non-O_2" pulps [7].

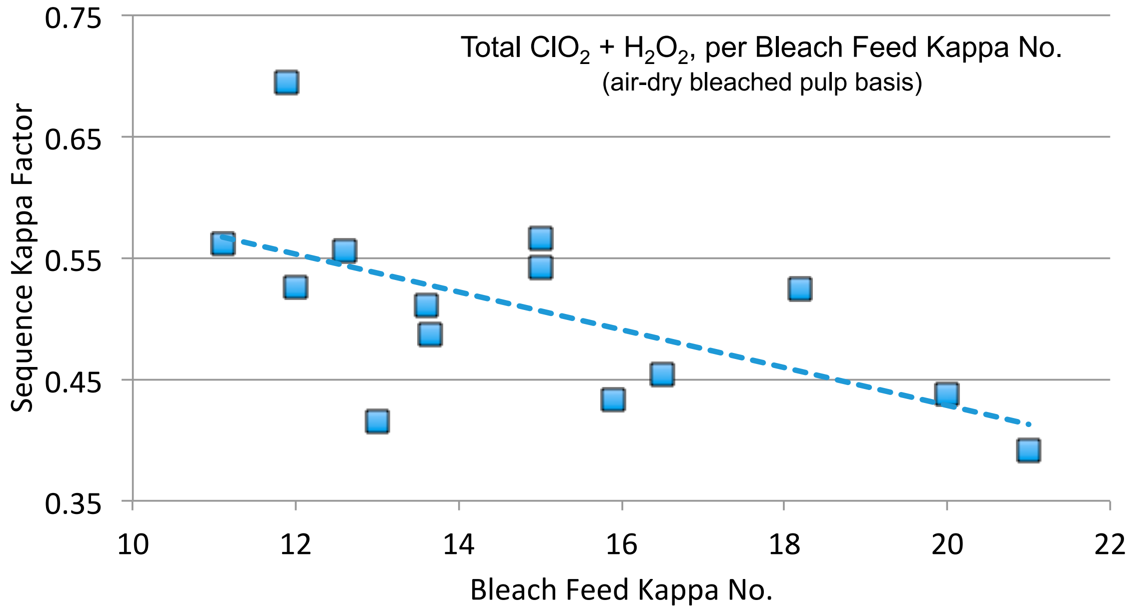

Figure 12: Oxidizing Chemical Use per Bleach Feed Kappa No. (Oxygen-Delignified Softwood)

Interestingly however there was no relationship between the *extent* of oxygen delignification and the relative chemical use, as shown in Figure 13; mills obtaining 50-60% lignin removal in the oxygen delignification stage operated with similar Sequence Kappa Factors as those running to 35-40% delignification. From this we can speculate that the *degree* of oxygen delignification does not impact the reactivity with chlorine dioxide significantly; instead it is the extent of delignification ahead of the bleach plant (digester plus oxygen delignification) which is the determining factor.

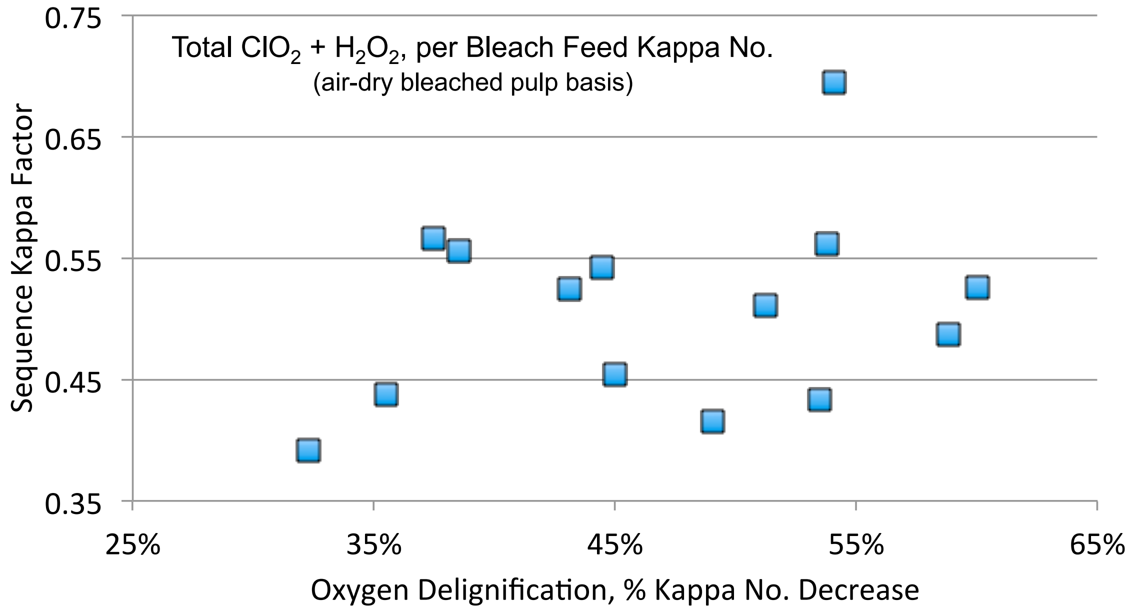

Figure 13: Impact of Oxygen Delignification on Bleaching Chemical Demand (Oxygen-Delignified Softwood)

PART 3: PRACTICES LEADING TO LOWER BLEACHING CHEMICAL USE

In order to further understand the best practices of North American bleach plants, the responses were divided into two groups, based on the Sequence Kappa Factor. The "low relative chemical consumption" mills are those with a Sequence Kappa Factor below the median for the group, while the "high relative chemical consumption" mills operate with a Sequence Kappa Factor above the median. The averages for these two data sets are presented in full in Table 1 in the Appendix; key parameters are shown in Table 2 below.

Table 2 – Relative Bleaching Chemical Consumption for Oxygen-Delignified Softwood Kraft Pulp Mills

Parameter	Low Relative Chemical Consumption Average	High Relative Chemical Consumption Average
Pre-O_2 Kappa No.	30.4	26.2
Bleach Feed Kappa No.	16.5	13.7
O_2 Delignification	45%	47%
D_0-stage Kappa Factor	0.22	0.32
Eop-stage NaOH, kg/ADMT	13.2	13.6
Eop-stage H_2O_2, kg/ADMT	3.5	1.5
Post-Eop Kappa No.	3.4	3.0
D1-stage ClO_2, kg/ADMT	9.2	8.6
D2-stage ClO_2, kg/ADMT	2.0	1.8
Final Brightness, % ISO	89.2%	88.7%
Total ClO_2, kg/ADMT	23.9	27.2
Total H_2O_2, kg/ADMT	4.1	2.4
Total NaOH, kg/ADMT	17.2	18.6
Sequence Kappa Factor	0.44	0.56

The low relative chemical consumption mills feed the bleach plant with a pulp that is on average 2.8 kappa units higher than the high chemical consumption mills, yet they use an average of 3.3 kg/ADMT less chlorine dioxide, and produce a higher brightness pulp.

Given that these are all different bleach plants, there are many factors involved when comparing mills, such as wood species, post-O_2 and bleach plant washing efficiency, bleach plant configuration and loading, and process control. But one relationship that is clear is the importance of a low D_0-stage Kappa Factor in achieving lower overall chemical use. Figure 14 clearly shows that bleach plants with a low Kappa Factor in the first chlorine dioxide stage tended to have a lower Sequence Kappa Factor as well. This was demonstrated in the previous survey as well [5], and is consistent with earlier studies on bleach plant optimization [8,9].

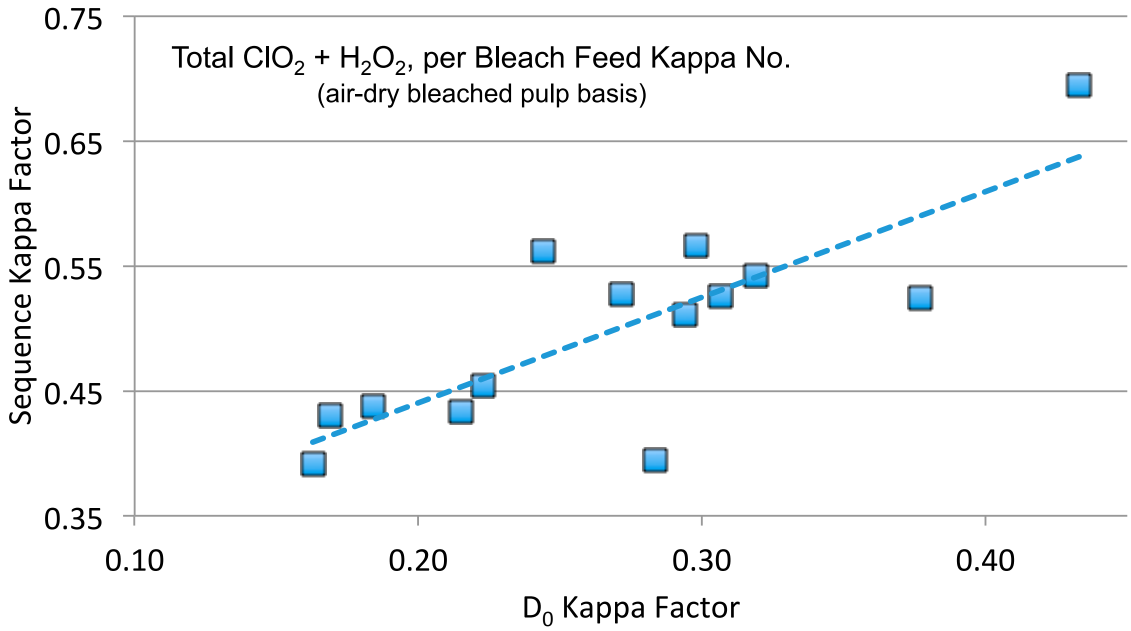

Figure 14: Impact of D_0-stage Kappa Factor on Total Bleaching Chemical Use (Oxygen-Delignified Softwood)

As noted in Table 1, the high relative consumption mills use almost 50% more chlorine dioxide in the first stage (on a Kappa Factor basis) compared to the low relative consumption mills. However this is not due to carryover; only one of the 14 respondents reported a high COD loading into the bleach plant. As well, the difference is not due to a shorter bleaching sequence, as both of the 3-stage bleach plants were in the "low" category, and the 5-stage bleach plants were split 4:3 between the "low" and "high" groups. And there was no apparent impact of wood species on bleaching chemical demand.

With regard to D_0-stage sensors and analyzers, the two groups were relatively similar; all but one of the respondents has an online kappa analyzer, and 11 of the 14 bleach plants have pre-tower optical and residual sensors. But the two groups use this equipment differently, as shown in Figure 15; the low relative consumption mills used their sensors and kappa analyzer in combination with each other, while the high relative consumption mills were more likely to use either compensated brightness alone, or Kappa Factor alone.

The low relative consumption mills also delignified to a higher post-Eop kappa no., 3.4 versus 3.0 for the high relative consumption mills. This is probably due in part to the higher bleach feed kappa no., but when combined with the significantly lower D_0-stage Kappa Factor, also serves to illustrate more optimal operation; *i.e.* high chemical-consuming bleach plants tend to over-bleach in the D_0 stage and so operate on the flat portion of the delignification curve.

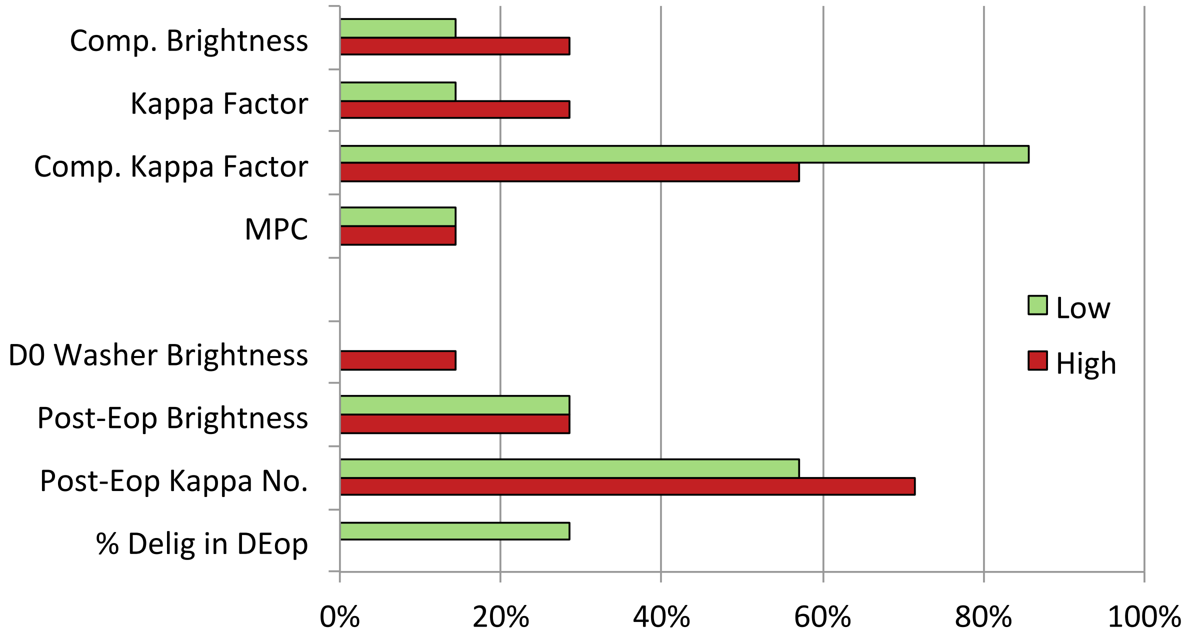

Figure 15: D_0-stage Process Control (Oxygen-Delignified Softwood)

Another significant difference between the two groups was the use of hydrogen peroxide. The low relative consumption mills used a total of 4.1 kg/ADMT H_2O_2 (3.5 kg/ADMT in the Eop stage), while the high relative consumption mills used an average of only 2.4 kg/ADMT total H_2O_2 (1.5 kg/ADMT in the Eop stage). This is a marked change from the previous survey, where the high relative consumption mills used substantially more peroxide in the Eop stage. It would appear that the low relative consumption mills are better-optimized with regard to their use of peroxide, while the high relative consumption mills now tend to use insufficient peroxide, at the expense of more chlorine dioxide.

There was no significant difference in the peroxide control strategy between the two groups; most mills still apply hydrogen peroxide at a fixed dosage, but 20% of the respondents are now controlling the peroxide charge based on either the incoming or target post-Eop kappa number.

Both groups operate the D1 stage to approximately the same average end-of-stage pH (pH 3.9 for the "low" group and pH 4.0 for the "high" group), but the range was quite broad, from a low terminal pH of 2.3 to a high of 5.0. More significantly perhaps, the low relative consumption mills have a lower ratio of D1-stage ClO_2 to the E1 kappa no. (2.7 kg/ADMT per kappa for the "low" mills, versus 3.3 kg/ADMT/kappa for the "high" mills). Previous studies have shown that a higher ClO_2-to-kappa ratio is often a result of poor Eop-stage washing, and carryover of Eop-stage filtrate into the D1 stage [10,11].

The low relative consumption mills also control the D1 stage differently, as shown in Figure 16. These mills are more likely to use a combination of the Eop-stage kappa no. and post-Eop brightness for chlorine dioxide control, and they are also more likely to use feedback control from the post-D1 brightness to adjust the ClO_2 charge.

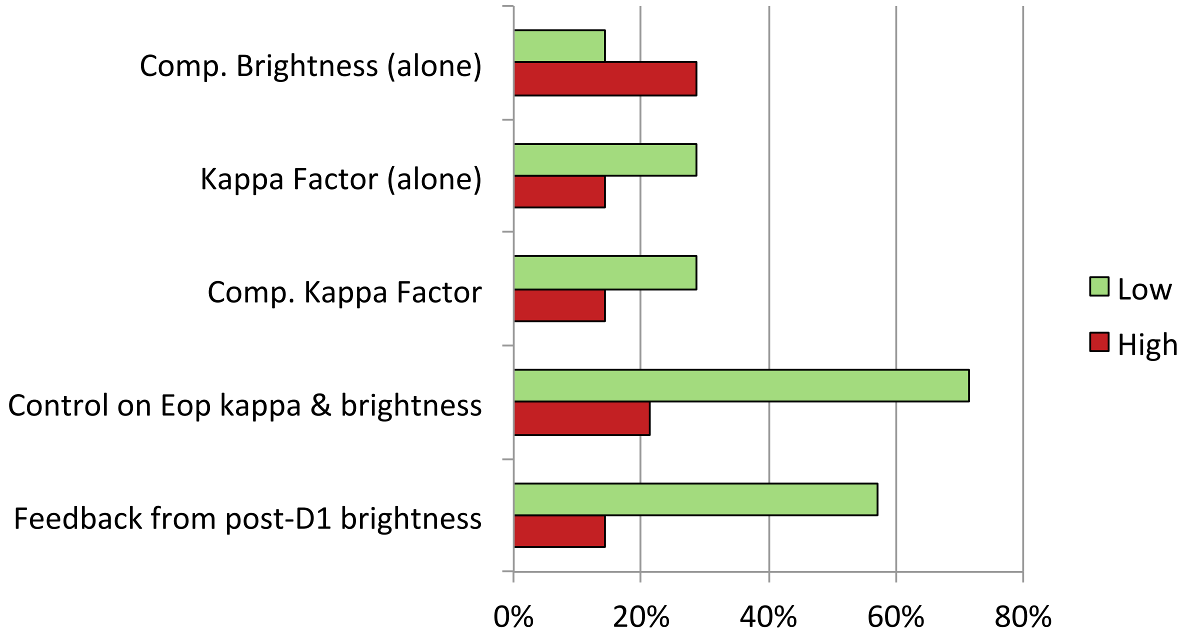

Figure 16: D1-stage Process Control (Oxygen-Delignified Softwood)

One final difference between the two groups is the operation of the final D2 stage. The low relative consumption mills run to a higher average end-of-stage pH, 5.7 versus 4.9 for the high relative consumption mills. This is a significant difference from the previous survey, when both groups operated the D2 stage much like a D1 stage, and likely reflects an improved understanding of the importance of pH in the final D stage [12,13], and an increased adoption of Near Neutral Bleaching [13].

In summary, "low relative chemical consuming" bleach plants producing oxygen-delignified softwood pulp:

- Feed the bleach with a pulp having a kappa no. 2.8 units higher than the "high relative consumption" bleach plants;
- Produce pulp with 0.5 points higher brightness;
- Use 3.3 kg/ADMT less chlorine dioxide, but 1.7 kg/ADMT more hydrogen peroxide;
- Operate with a significantly lower D_0-stage Kappa Factor (0.22 versus 0.32);
- Are more likely to control the D_0 stage using an online kappa analyzer in combination with optical and residual sensors;
- Are more likely to control the D1 stage using a combination of post-Eop kappa no. and post-Eop brightness, with feedback from the post-D1 brightness;
- Have a lower ratio of D1-stage chlorine dioxide to the Eop kappa number;
- Operate with a higher pH in the D2 stage.

ACKNOWLEDGMENTS

The authors would like to thank all of the pulp mill personnel who took the time to complete this survey. Only through their efforts was this report made possible.

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Table 1: Summary of Oxygen-Delignified Softwood Kraft Pulp Bleaching "Best Practices" – Group Averages

	Relative Bleaching Sequence Chemical Consumption*	
	Low	**High**
Final Machine Brightness, % ISO	**89.2**	**88.7**
Number of Mills	**6**	**7**
Number of Bleach Plants	**7**	**7**
Oxygen Delignification		
Digester Kappa No.	31.2	27.4
Pre-O_2 Kappa No.	30.4	26.2
COD entering O_2 stage, kg/ADMT	26	137
Alkali charge, kg/ADMT eq. NaOH (total)	20.3	18.8
Oxygen charge, kg/ADMT (total)	15.0	20.8
Post-O_2 Kappa No.	16.7	13.7
O_2 Delignification, %	45%	47%
First ClO_2 Stage (D_0)		
Kappa No. entering D_0 Stage	16.5	13.7
COD Carryover, kg/ADMT	9.0	15.0
Na_2SO_4 Carryover, kg/ADMT	9.0	4.8
Chlorine Dioxide, kg/ADMT	13.3	16.9
D_0 Kappa Factor	0.22	0.32
Retention Time, min.	69	44
Temperature, °C	64	61
Terminal pH	2.3	2.6
First Extraction Stage (Eop)		
Sodium Hydroxide, kg/ADMT	13.2	13.6
Ratio, Eop NaOH to D_0 ClO_2	0.98	0.86
Oxygen, kg/ADMT	4.2	3.2
Hydrogen Peroxide, kg/ADMT	3.5	1.5
Retention Time under pressure, min.	17	12
Total Retention Time, min.	77	52
Temperature, °C	77	78
Terminal pH	10.5	10.3
Post-Eop Kappa No.	3.4	3.0
Post-Eop Brightness, % ISO	62.7	56.7

* Ranking by Sequence Kappa Factor, based on all chlorine dioxide and hydrogen peroxide used in the bleach plant. Does not include oxygen delignification stage.

Table 1: Summary of Oxygen-Delignified Softwood Kraft Pulp Bleaching "Best Practices" – Group Averages (continued)

	Relative Bleaching Sequence Chemical Consumption*	
	Low	**High**
Final Machine Brightness, % ISO	**89.2**	**88.7**
Number of Mills	**6**	**7**
Number of Bleach Plants	**7**	**7**
First ClO_2 Brightening Stage (D1)		
Chlorine Dioxide, kg/ADMT	9.2	8.6
Retention Time, min.	154	161
Temperature, °C	77	73
Terminal pH	3.9	4.0
D1 Brightness, % ISO	84.6**	83.9
3-stage bleach plants	2	0
4-stage bleach plants	1	4
5-stage bleach plants	4	3
Second Extraction Stage (E2/n)		
Sodium Hydroxide, kg/ADMT	4.9	5.5
Hydrogen Peroxide, kg/ADMT (when used)	1.6	1.2
Retention Time, min.	58	46
Temperature, °C	75	75
Terminal pH	10.2	9.6
Full Extraction (E2) stage	4	3
Short Neutralization ("n") stage	1	2
No Extraction between D1 and D2	0	2

* Ranking by Sequence Kappa Factor, based on all chlorine dioxide and hydrogen peroxide used in the bleach plant. Does not include oxygen delignification stage.

** Note that average "low" D1 brightness is skewed by the two 3-stage bleach plants; excluding these, the average D1-stage brightness would be 82.9% ISO.

Table 1: Summary of Oxygen-Delignified Softwood Kraft Pulp Bleaching "Best Practices" – Group Averages (continued)

	Relative Bleaching Sequence Chemical Consumption*	
	Low	**High**
Final Machine Brightness, % ISO	**89.2**	**88.7**
Number of Mills	**6**	**7**
Number of Bleach Plants	**7**	**7**
Second ClO_2 Brightening Stage (D2)		
Chlorine Dioxide, kg/ADMT	2.0	1.8
Retention Time, min.	148	192
Temperature, °C	78	77
Terminal pH	5.7	4.9
D2 Brightness, % ISO	89.6	88.7
Total Bleaching Chemical Use		
Bleach Feed Kappa No.	16.5	13.7
Chlorine Dioxide, kg/ADMT	23.9	27.2
Sodium Hydroxide, kg/ADMT**	17.2	18.6
Hydrogen Peroxide, kg/ADMT	4.1	2.4
Oxygen, kg/ADMT	4.2	3.2
Sequence Kappa Factor***	0.44	0.56

* Ranking by Sequence Kappa Factor, based on all chlorine dioxide and hydrogen peroxide used in the bleach plant. Does not include oxygen delignification stage.

** Includes sodium hydroxide used for pH control in the D1 and D2 stages.

*** Sum of all chlorine dioxide and all hydrogen peroxide used in the bleach plant, all expressed as % equivalent chlorine on pulp, and divided by the bleach feed kappa number.