New approaches in bleaching of recycled fibers

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ABSTRACT

Investigation of chlorine free chemicals in bleaching recovered paper furnishes aimed at achieving higher brightness values necessary for high-quality writing or printing papers or tissue products. Removing transition metal ions and destroying catalysts in a preceding hot acid treatment enhances the efficiency of peroxide or (OP) treatments. (OP) stages are superior to P stages especially for optical homogeneity. Ozone is a powerful brightening agent for recovered paper furnishes with no or low mechanical fiber. In combination with a final P or FAS stage, a brightness of 85% ISO is possible. The same brightness level can result from (OP), FAS, or (OP) P bleaching of wood-free recovered papers such as colored ledger. A three-stage sequence of (OP), P, and FAS can brighten recovered paper to approximately 75% ISO.

Application:

Various bleaching techniques can improve the brightness of high quality papers made from recovered paper.

Existing recovered paper grades do not allow using a standardized stock preparation and bleaching procedure. Papers differ substantially from each other in fiber composition, content and type of fillers, inks, dyes, and impurities. Stock preparation depends on the quality specifications of a given paper product.

Intensive mechanical and physico-chemical pulp cleaning should precede recovered paper bleaching. Before bleaching, it is necessary to remove as many impurities as possible. The recovered paper bleaching operation integrated in a stock preparation plant therefore usually consists of pulping, cleaning, coarse and fine prescreening, flotation, post screening, dispersing, bleaching, and post flotation.

This is not a fixed treatment scheme. It can vary from mill to mill depending on the quality of the raw material and the desired final paper product. Sometimes there is a second or even a third flotation stage in treatment plants for high grades such as substitutes for chemical pulp that require high brightness and cleanliness.

Bleaching is only one step in a complex stock preparation system. It deserves considerable attention, however, because it is the key to using recovered paper in high-quality paper products. The most common recovered paper bleaching practice uses the lignin retaining bleaching chemicals hydrogen peroxide, sodium hydrosulfite (sodium dithionite), and formamidine sulfinic acid (FAS) ($HO_2S\text{-}CNH\text{-}NH_2$). In a standard operation, bleaching of old newsprint (ONP) and old magazines (OMG) for the production of newsprint uses one stage. The addition of 0.5–1.0% peroxide to the pulping stage does not accomplish the optimal parameters of peroxide bleaching. The primary function is to prevent alkaline yellowing. The required stock brightness for newsprint production is approximately 60% ISO.

A single peroxide or hydrosulfite stage after flotation may increase brightness up to 64% ISO. It is possible to conduct such a bleaching stage in a tower using high consistency (HC) or in a tube. Chemical mixing can be done in a medium consistency (MC) pump or in a disperser or kneader *(1)*. A disperser or kneader has two functions—mixing and dispersing of contaminants such as ink. An obvious question arises. Is peroxide or hydrosulfite a better brightening agent? In cases where there has been a peroxide treatment before the main bleaching operation, hydrosulfite may be the better alternative because the brightening response of the recovered paper is better. Since rapidly oxidizable chromophoric structures have reacted in the previous peroxide treatment, hydrosulfite can efficiently reduce quinoid structures or dyes. In addition, peroxide requires an effective stabilization especially in recovered paper bleaching.

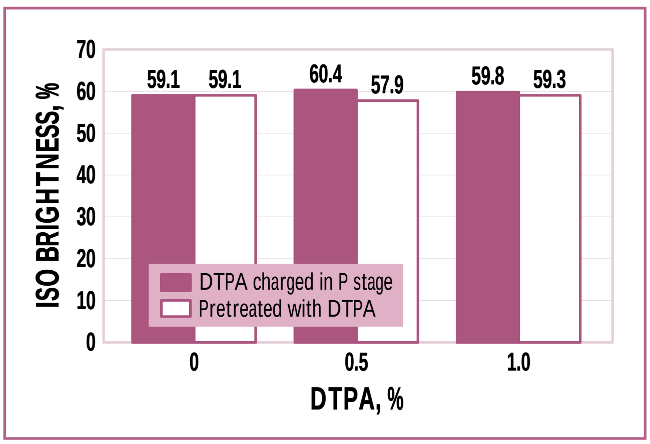
FAS is another very efficient bleaching agent for reducing. It is less sensitive than hydrosulfite to air oxidation or decomposition by transition metal ions. The main disadvantage is its high price compared to other bleaching agents.

For more efficient recovered paper bleaching, two stages are necessary. Different combinations are in commercial use. Most installations use an oxidative treatment in the first stage. The second stage runs with hydrosulfite or FAS. Sometimes the use of FAS is more effective and has the advantage that no additional acidification is necessary. We recommend stock dilution and rethickening between two bleaching stages. The dilution step should preferably combine with a post flotation or washing stage followed by dewatering on a double wire press.

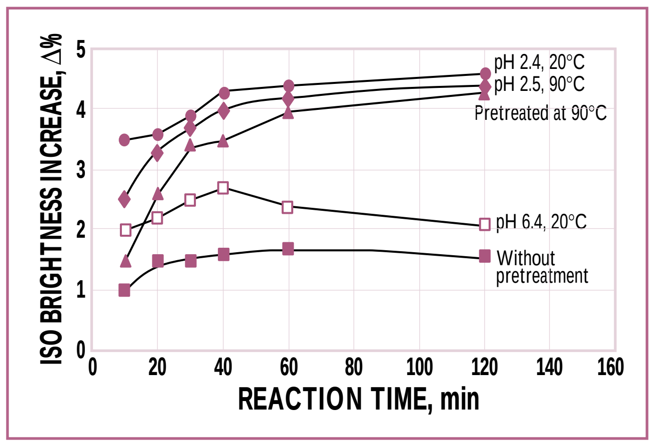
A separation between bleaching stages avoids chemical carryover with concomitant degradation of the

Bleaching agents and sequences	Brightness gain, wood free, Δ % ISO	Brightness gain, wood containing Δ % ISO	Process parameters	Waste paper grade	Effects	Remarks
Hydrogen peroxide:			0.5–3%		No deinking or cleaning effect	Silicate required
P	3–15	2–12	0.5–2% NaOH		No dye removal	with stabilizers
P Y	8–20	10–15	40–95°C	All grades	High doses cause strength losses in wood free fibers	at high temp.
P FAS	8–20	10–17	Tower (MC) Disperser (HC) Pulper (MC/HC)			Applicable in dispersers
Sodium dithionite:			0.3–1% 60–95°C	All grades	Removes dyes Effective bleaching of wood free fibers	Presence of air and peroxide carryover must be avoided
Y	3–15	2–7	Tower (MC)			
Y P	8–17	-	Disperser (HC)			
Formamidine sulfinic acid:			0.3–1% 70–95°C	All grades	Reduces dyes more effectively than sodium dithionite	Insensitive to O_2 and very heat resistant
FAS	6–15	3–10	0.2–0.5% NaOH			
FAS P	8–18	8–12	Disperser (HC) or pipe (MC)			Triple price

I. Conventional bleaching of secondary fibers



1. Effect of DTPA on peroxide bleaching of mixed recovered paper with high filler content



2. Influence of pretreatment conditions on the brightness increase in a P stage using 3% H_2O_2 with 40/60 ONP/OMG

second bleaching agent. It also improves cleanliness by washing dissolved particles from the first bleaching stage and by removal of residual inks, dyes, and other disturbing substances. Besides enhanced bleaching efficiency, this practice provides the best opportunity for separation of the water loops.

There is a strong tendency toward higher consistency particularly in recovered paper bleaching. Peroxide stages are in operation today that work at 30% consistency. This improves the brightness response, saves energy and alkali, and reduces the COD load. The trend should continue so that oxidative and reducing bleaching will use high consistency. Peroxide bleaching will even use ultra high consistency.

Table I presents information by Gehr *(2)* showing a summary of recovered paper bleaching with peroxide, hydrosulfite, and FAS. The remarks in the table refer to the first bleaching agent. Note that the brightness increase strongly depends on the raw material quality.

NEW BLEACHING SEQUENCES

The brightness gain obtainable in conventional recovered paper bleaching has limitations when the available recovered paper qualities become poorer. Then it is necessary to upgrade the material for the production of high-quality papers. There is a need for more efficient bleaching sequences to meet the general target of enhanced recovered paper recycling rates.

There is a certain potential for improving the action of peroxide in recovered paper bleaching. In contrast to virgin pulps, the transition

Bleaching stage	Kappa no.	Brightness, ISO	Ash content, %	Cu, ppm	Fe, ppm	Mn, ppm	Mg, ppm	Ca, ppm
Unbleached	69.7	63.3	9.9	9.9	256	7.3	651	9980
A	69.9	64.3	6.9	7.2	240	2.1	183	28
A (OP)	66.9	68.0	7.0	6.2	231	1.7	1170	16

II. Data for the ONP/OMG pulp

Bleaching sequence	DCM extractives, %	Dot counts	L*	a*	b*	Brightness, % ISO
(OP)	0.60	47.1	86.9	0.1	6.9	63.8
(OP) P_2	-	-	88.9	-0.3	6.5	67.0
(OP) P_2 FAS	0.41	-	90.2	-0.7	5.6	70.5
P_1	0.79	68.8	86.5	0	6.6	63.1
P_1 P_2	-	-	88.6	-0.2	6.7	66.3
P_1 P_2 FAS	0.65	-	89.4	-0.5	6.1	68.9

(Units for dot counts, L*, a*, and b* are 1 x $10^3/m^2$)

III. Comparison of (OP) vs. P bleaching for a flotated and acid pretreated ONP/OMG furnish with high mechanical pulp content with kappa no. 105

metal content in recovered paper furnishes can be high. Some post-consumer grades have heavy contamination with catalysts. Transition metals and catalysts both cause fast decomposition of peroxide. The standard metal complexing agent in peroxide bleaching is sodium silicate. When using hard water or in the presence of $CaCO_3$ filler, sodium silicate can cause trouble in papermaking *(3, 4)*. Calcium or magnesium silicates precipitate and cause scaling problems on the paper machine. In addition, the anionic silicate ions may impair the action of different cationic additives in papermaking such as retention aids or wet-strength aids. Figure 1 shows that the application of diaminetriethylenepentaacetic acid (DTPA) in peroxide bleaching of mixed recovered paper or in a pretreatment stage has practically no effect. The heavy metal and filler contents of the grades were too high. The complexing agents chelated other metal ions on a stoichiometric basis *(5)*. One therefore should restrict the use of amino polycarbonic acids to peroxide bleaching of wood free grades with no filler content or low filler content.

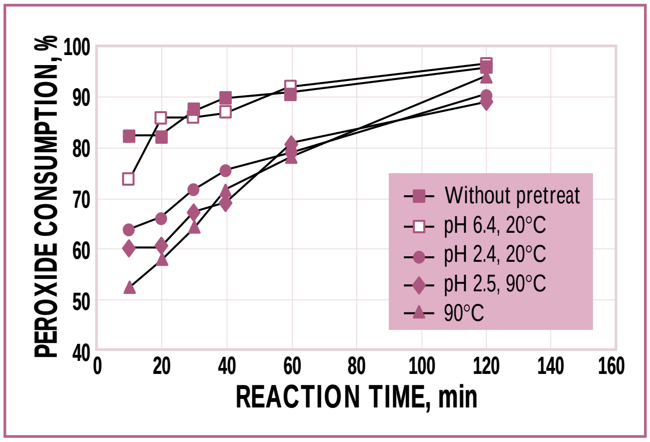
An acid treatment can perform heavy metal removal. We subjected a 40/60 ONP/OMG furnish to different pretreatments. The furnish was prepared in a stock preparation mill with 1% H_2O_2 added in the pulper. After precleaning, preflotation, and dispersing, the stock passed a post flotation stage. The kappa number of the stock was 70. Using this value, it is possible to estimate a content of approximately 40% mechanical fiber. Table II shows the metal contents of the pulp before and after acid treatment. In this table, the conditions for the A stage were an entering pH of 2.5, a temperature of 20°C, a time of 60 min, and a consistency of 3%. For the (OP) stage, the H_2O_2 was 1.5%, the NaOH was 1%, and the $MgSO_4$ was 0.75%

The acid treatment reduced the calcium content of the stock drastically. It meant a large part of the filler content was calcium carbonate. The ash content decreased by 3%. The content of magnesium ions also decreased considerably. It was necessary to compensate for the loss by addition of $MgSO_4$ in the (OP) stage to reconstitute the protecting effect of magnesium ions against peroxide degradation. The acid stage had virtually no effect on the iron content. Previous work showed that iron ions have a strong fixation to the fibers and that an acid treatment cannot mobilize them *(6)*. The copper content remained high also. There was a high degree of removal of the manganese ions—the most harmful to peroxide bleaching.

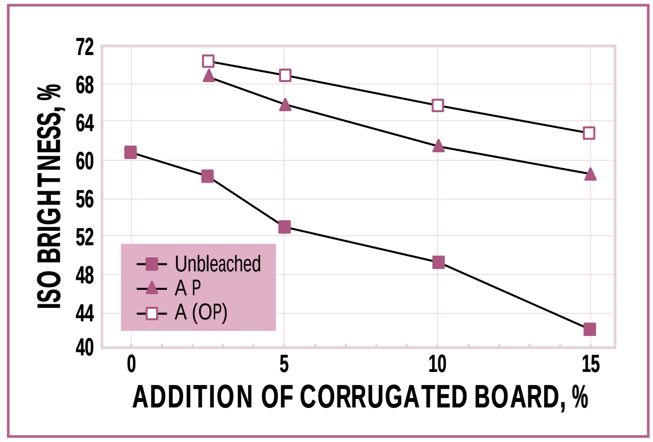
The efficiency of the acid stage depends on the conditions applied. Figures 2 and 3 show that one should adjust the pH of such a treatment to 2.5 or less. At temperatures over 70°C, there can be simultaneous destruction of catalysts.

P VERSUS (OP) BLEACHING

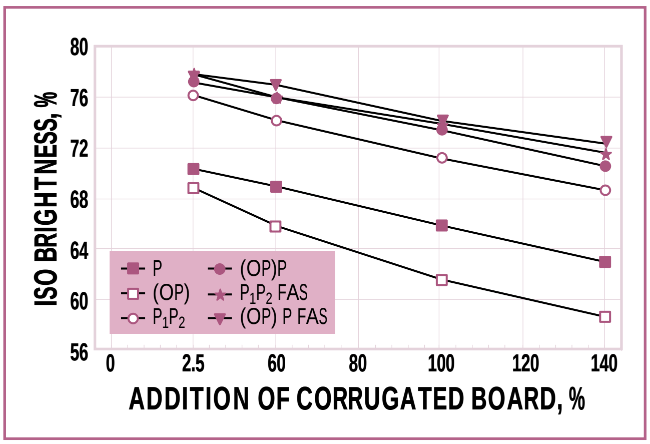
We compared the efficiency of P and (OP) treatments for two different recovered paper grades. The first was an ONP/OMG pulp with high mechanical pulp content of kappa no. approximately 105. The second was a wood-free material consisting of 50% white ledger (WL) and 50% colored ledger (CL) with a kappa



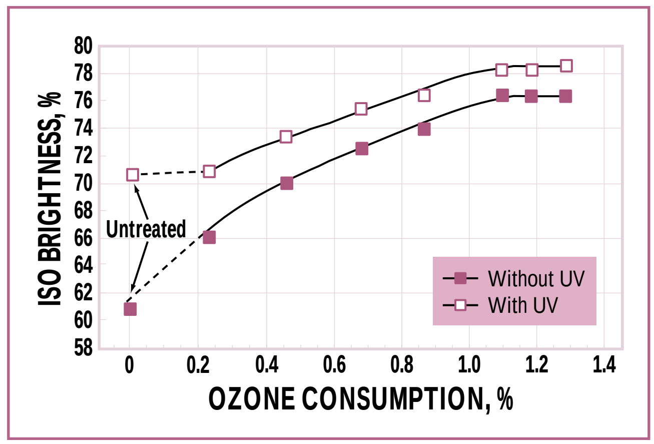
3. Peroxide consumption during P stage showing dependence on the pretreatment conditions using 3% H_2O_2 with 40/60 ONP/OMG



4. Influence of corrugated board addition to WL/CL pulp on brightness after P or (OP) bleaching



5. Influence of corrugated board addition to WL/CL pulp on brightness after multistage sequences with P or (OP) as first stage



6. Ozone bleaching of WL/CL pulp with kappa no. 6.5 showing brightness measurement with and without UV luminance

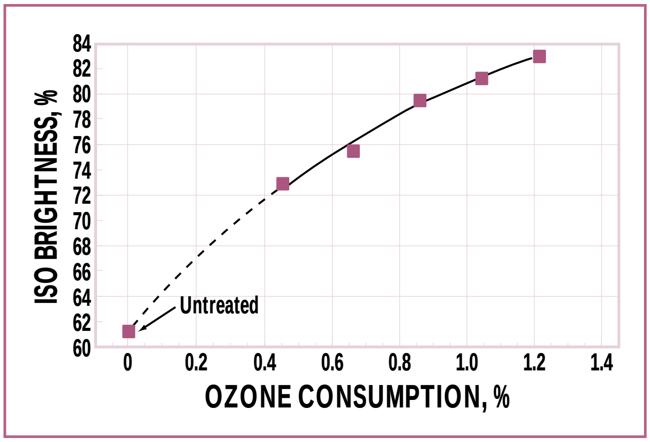
number of 6.5. Both pulps used identical conditions of peroxide charge, temperature, and reaction time. In both cases, the difference in brightness between P and (OP) treatment was less than one brightness point in favor of the (OP) treated pulp.

We subjected the wood-containing recovered paper furnish to different bleaching sequences with an initial P or (OP) stage. With more intensive bleaching, the brightness advantage of the (OP) treated sample became greater. In addition, Table III shows that (OP) bleaching reduced the content of dirt and dichloromethane (DCM) extractives using TAPPI T 204 om-88 more efficiently. In the table, the P_1 stage has 1% H_2O_2, 1% NaOH, and 0.75% $MgSO_4$; the P_2 stage has 2% H_2O_2, 1% NaOH, and 3% Na_2SO_3; and the FAS stage has 0.7% FAS and 0.35% NaOH.

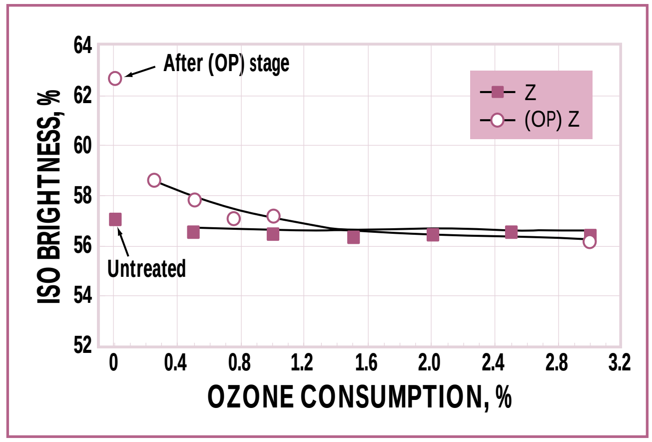
A general problem in preparation of secondary pulp from mixed office recovered paper (MOW) is the presence of unbleached kraft pulp fibers with high lignin content. The mottled appearance initiated by these brown fibers becomes more intensive after bleaching. We mixed different amounts of corrugated board in the WL/CL secondary pulp and conducted P or (OP) stages alternatively after acid pretreatment as Fig. 4 shows.

Without addition of corrugated board to the furnish, there was only a small advantage in brightness for the (OP) treatment. With increasing amounts of unbleached kraft fibers, the difference became more distinct, however. In addition, the yellowness decreased more. At 15% addition of corrugated board, the yellowness decreased from 13.1 to 9.6 in the (OP) stage. It only decreased 1.8 units in the P stage.

The superiority of an (OP) treatment was also apparent after multistage bleaching. Figure 5 shows that the gap in brightness between (OP) and P bleaching became smaller with more stages.



7. Ozone bleaching of MOW pulp with kappa no. 8.5



8. Effect of ozone consumption on brightness of untreated or (OP) prebleached ONP/OMG pulp with high content of mechanical fibers at kappa no. 105

<table>
<tr><th>Bleaching agents and sequences</th><th>Brightness gain, wood free, Δ % ISO</th><th>Brightness gain, wood containing, Δ % ISO</th><th>Process parameters</th><th>Recovered paper grade</th><th>Effects</th><th>Remarks</th></tr>
<tr><td>Oxygen:</td><td></td><td></td><td>ca. 1–3%
1–5% NaOH
30–150 min
70–120°C</td><td>Wood containing pulps only in presence of H_2O_2 to prevent alkaline yellowing</td><td>Improves optical homogeneity by removal of ink and mottled fibers
Adhesives and waxes
Waxes are partially dissolved
Higher fiber flexibility and strength by delignification</td><td>Depends on raw material composition
Strength improvement possible
Very effective in combination with peroxide to save peroxide</td></tr>
<tr><td>O</td><td>2–12</td><td>-</td><td></td><td></td><td></td><td></td></tr>
<tr><td>(OP)</td><td>4–20</td><td>4–11</td><td></td><td></td><td></td><td></td></tr>
<tr><td>(OP) P FAS | Y</td><td>6–20</td><td>9–17</td><td></td><td></td><td></td><td></td></tr>
<tr><td>Ozone:</td><td></td><td></td><td>0.5–4%
Primarily HC technique</td><td>No or only low content of mechanical pulp, otherwise no brightness gain</td><td>Increase or loss in strength depending on the raw material composition
High brightness gain at high content of unbleached kraft fibers
No deinking effect</td><td>The most effective bleaching agent for wood free grades
Effective removal of dyes, brown fibers, and optical brighteners</td></tr>
<tr><td>Z</td><td>5–20</td><td>-</td><td></td><td></td><td></td><td></td></tr>
<tr><td>Z P, Z P Y</td><td>6–25</td><td>_</td><td></td><td></td><td></td><td></td></tr>
<tr><td>(OP) Z FAS | Y</td><td>up to 35</td><td>-</td><td></td><td></td><td></td><td></td></tr>
</table>

IV. Nonconventional bleaching of secondary fibers

APPLICATION OF OZONE IN RECOVERED PAPER BLEACHING

Ozone treatment of pulps has two goals:

- Delignification and concomitant bleaching of chemical pulps
- Improvement of the bonding potential of the fiber surfaces by introduction of hydrophilic groups.

The application of ozone to wood free furnishes improves brightness considerably. Simultaneously ozone degrades cellulose and reduces fiber strength. In contrast, the reaction of ozone with mechanical pulp improves the strength of the fiber network. Pulp brightness decreases due to formation of quinoid lignin structures. The divergent effects of ozone treatment on chemical and mechanical pulps clearly show the dilemma of ozone application for bleaching of mixed recovered paper grades.

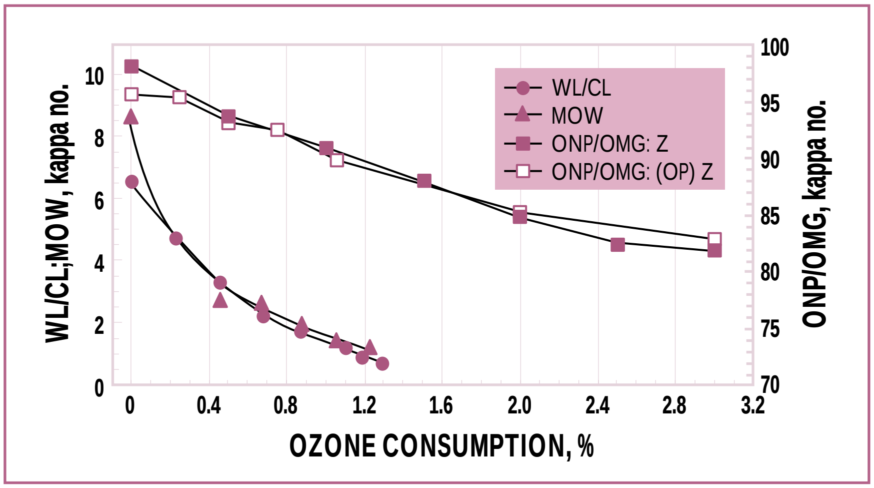
In our trials, we subjected two wood free papers and a wood containing recovered paper to ozone treatment—the WL/CL furnish with kappa no. 6.5, a MOW pulp having kappa no. 8.5, and the ONP/OMG mixture consisting of approximately 65% mechanical pulp and 35% chemical pulp with kappa no. 105.

The mixed WL/CL pulp contained optical brighteners. This is evident from Fig. 6 showing a difference of 10 points between brightness measurement with or without inserting an ultraviolet (UV) cutoff filter. With increasing ozone charge, the difference in brightness with and without UV luminance became smaller. This indicates that ozone was destroying fluorescence components. At an ozone charge of 1.1%, the brightness increased by only 8 points when measured with UV luminance. It increased by 15 points without UV luminance. The MOW at kappa no. 8.5 showed an even better response to ozone treatment. It was easily bleached to a brightness over 80% ISO. At 1% ozone consumption, Fig. 7 shows that the brightness increased approximately 20 points.

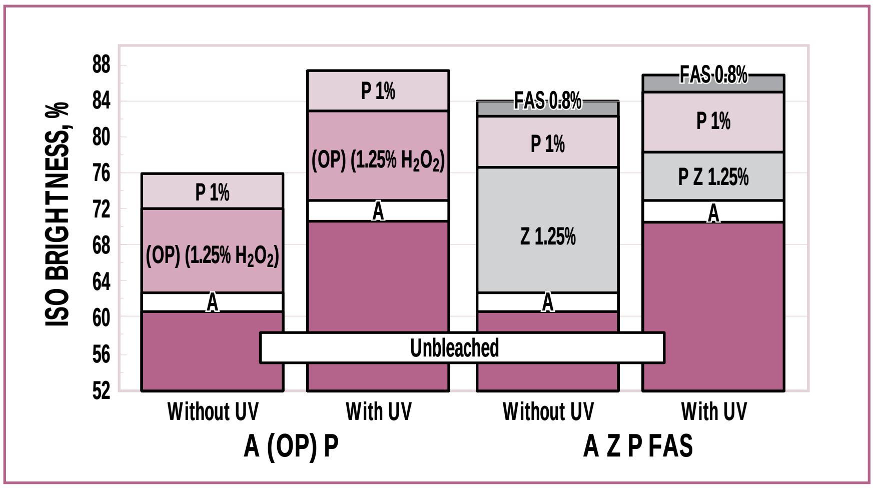
Figure 8 illustrates the effect of different ozone dosages on brightness of the ONP/OMG pulp with a high kappa no. of 105. As outlined before, ozone treatment of wood containing recovered paper furnishes leads to a loss in brightness. The brightness decrease of two points already took place at low ozone consumption. Further ozone uptake did not affect brightness. Although an initial (OP) stage improves pulp brightness, the brightness decreased again in the subsequent Z stage with increasing ozone consumption. Figure 8 shows that it finally reached the level of the directly ozone treated pulp.

Figure 9 shows that ozone treatment reduces the lignin content very effectively. For wood-containing recovered paper furnishes, however, high ozone charges are necessary to attain a significant kappa number reduction. Even such an extensive delignification does not give a brightness gain, because brightening in an ozone treatment does not start until reaching a very low content of residual lignin. We therefore can conclude from our experience that ozone bleaching of recovered paper is only worthwhile when the content of mechanical fibers does not exceed approximately 10%. In particular, ozone treatment can be successful when applied to improve optical homogeneity with a furnish containing some unbleached kraft fibers.

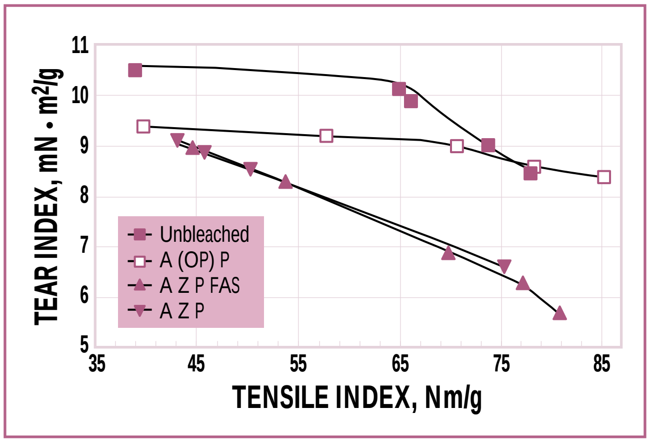
Table IV is also data by Gehr *(2)* that summarizes the potential applications of oxygen and ozone in recovered paper bleaching. The remarks again refer to the first bleaching agent, and the brightness strongly depends on raw material quality. The brightness improvement attained by application of different bleaching sequences depends primarily on the composition of the recovered paper, the stock preparation system, and on the bleaching conditions applied. In the presence of mechanical fibers in the furnish, one must use oxygen in combination with peroxide to obtain a brightening effect. Ozone is the most powerful bleaching agent for secondary fibers when the mechanical fiber content is low. It effectively removes the mottled appearance caused by brown fibers and can destroy a variety of organic dyes.



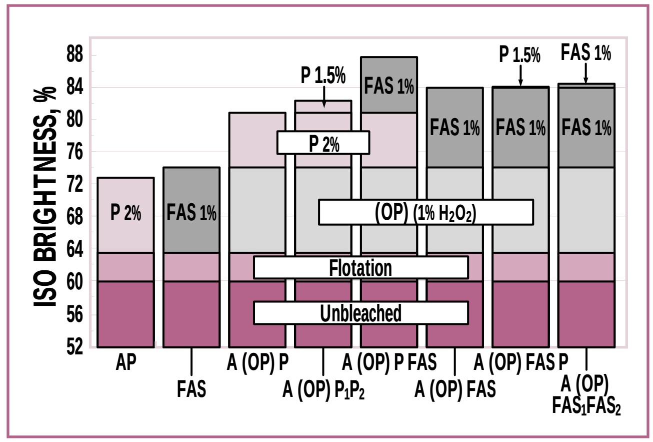
9. Ozone delignification of recovered paper grades with low or high lignin content



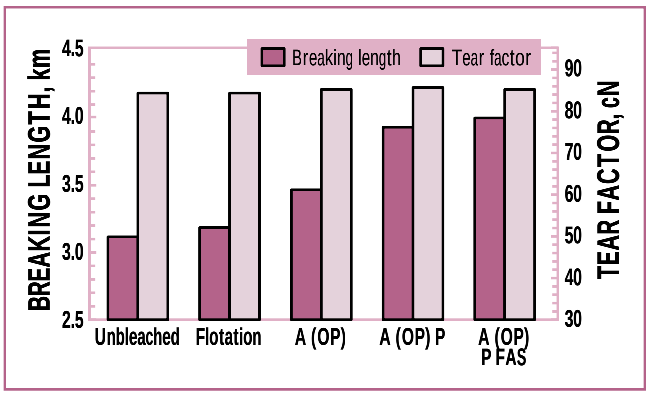
10. Multistage bleaching of WL/CL pulp



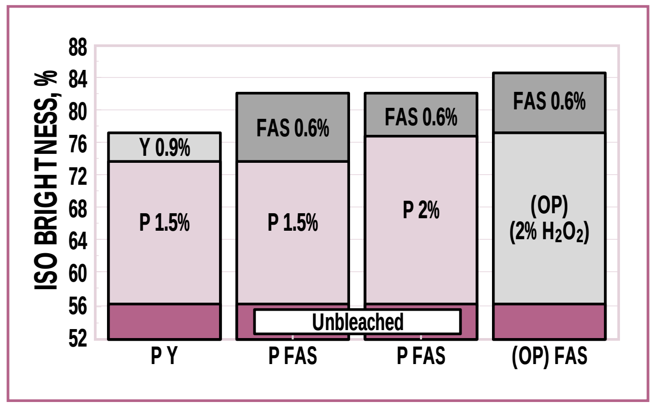
11. Tear vs. tensile plot for WL/CL pulp showing unbleached and different bleaching sequences



12. Multistage bleaching of an ONP/OMG furnish with high content of mechanical fibers at kappa no. 105



13. Strength development during (OP) P FAS bleaching of an ONP/OMG furnish at kappa no. 105



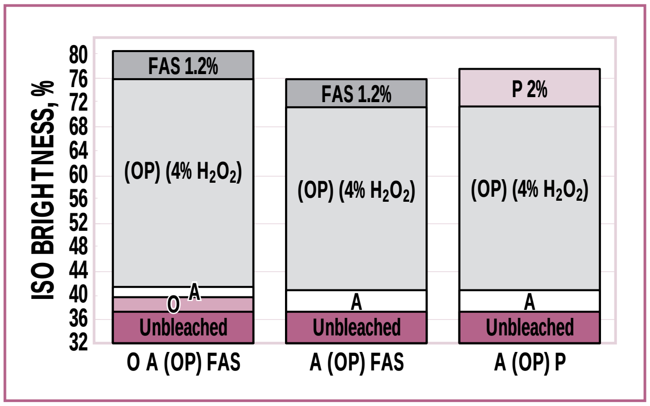
14. Bleaching of CL

POTENTIAL BLEACHING SEQUENCES FOR DIFFERENT RECOVERED PAPER GRADES

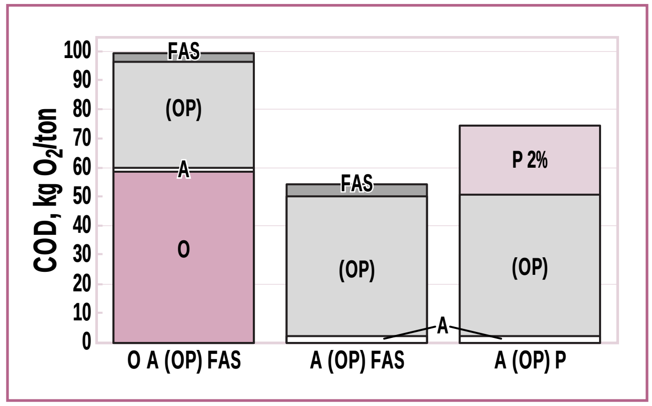
We subjected different recovered paper grades to various bleaching sequences. Figure 10 shows the bleaching results for the wood free 50/50 WL/CL furnish with kappa no. 6.5. With a moderate ozone charge, one can achieve a substantial increase of fiber brightness. Using brightness measurement with UV luminance, it becomes evident that ozone like sodium hypochlorite destroys fluorescent brighteners. This is necessary for use in food packaging in the United States and is helpful for control of color of the final paper product. Compared to ozone treatment, an (OP) stage is less effective in fiber brightening, but the optical brighteners are not destroyed. The higher brightness gain measured with UV luminance indicates that an (OP) stage even enhances the residual fluorescence. A peroxide treatment as second bleaching stage is significantly more effective after the initial Z stage. A final FAS stage after Z P bleaching has only a limited effect due to destruction of dyes in the preceding ozone treatment and the absence of mechanical fibers.

Figure 11 shows the strength properties of the recovered paper furnish. The (OP) P sequence reduced tear resistance at unchanged tensile strength. Ozone bleaching reactivates the bonding ability of the fiber surface to some extent. The tensile strength therefore could remain at the level of the unbleached recovered paper. Serious fiber damages had obviously occurred as indicated by the reduced tear strength.

The mixture of ONP/OMG with 65% mechanical pulp content at kappa no. 105 also underwent bleaching in different sequences. The brightness after post flotation was 59% ISO. An (OP) stage applying 1% peroxide gave a brightness of 66% ISO. The same result occurred with 1% FAS. A single P stage with 2% H_2O_2 charge gave lower brightness. Peroxide was also less effective than FAS when applied in the second stage after (OP) treatment. Two stage (OP) FAS bleaching improved brightness by 13 points. Figure 12 shows that the highest brightness in three stages, however, occurred in an (OP)



15. Bleaching of recycled LPB in different sequences



16. COD loads for bleaching of recycled LPB

KEYWORDS

Acid treatment, bleaching, bogus boards, bogus papers, hydrogen peroxide, mixed waste, multistage process, optical brighteners, oxygen, ozone, reclaimed fibers, sodium dithionite, sulfinic acids, waste papers.

P FAS sequence due to the enhanced brightening effect of FAS in the third stage.

Figure 13 shows the strength development during (OP) P FAS bleaching of the ONP/OMG furnish. With more intensive bleaching, the tensile strength improves while the tear strength remains constant. This strength performance is typical for pulps with high mechanical fiber content.

As Fig. 10 shows, it is possible to upgrade a wood-free recovered paper furnish to high brightness. At good optical homogeneity, such recycled pulp can substitute for virgin fibers in various products.

We also subjected two other recovered paper qualities with no fiber content or low mechanical fiber content to multistage bleaching to reach a high brightness level. The first furnish was 100% CL with a starting brightness of 56% ISO and a kappa number of approximately 20. P or (OP) bleaching charging 2% peroxide in both cases improved brightness by 20 and 21 points, respectively. With 0.6% FAS in a second stage, 83% or almost 85% ISO values were possible. The application of hydrosulfite instead of FAS was less efficient in color stripping as Fig. 14 shows. The second furnish was recycled liquid packaging board (LPB) with 37.3% ISO brightness and kappa no. 40. Figure 15 shows the brightness improvements obtained in three different bleaching sequences. The first bleaching sequence started with an oxygen delignification. Despite the high alkali charge of 4% applied in this stage, the kappa number only reduced by only 15 units and brightness improved only marginally. In addition, the O stage led to a high yield loss of 6% and generated a high specific COD load of approximately 60 kg O_2/ton. The bleaching yield for the O A (OP) FAS sequence was only 90%, and the total COD load exceeded 100 kg O_2/ton as Fig. 16 shows. Such a sequence enables a high brightness increase over 80% ISO, however. This is mainly due to the extreme brightening effect of the (OP) stage. Without oxygen delignification, a brightness of 75% ISO was possible at a bleaching yield of 93% and a much lower COD load of 60 kg O_2/ton for the whole sequence. A final P stage instead of the FAS treatment resulted in brightness two points higher at the expense of a considerably higher COD load.

CONCLUSIONS

Summarizing the work and observations above, it is possible to draw the following conclusions:

- Besides more efficient stock preparation systems, a further increase for recovered paper recycling rates requires improved bleaching technology.
- Catalysts that decompose peroxide frequently contaminate postconsumer recovered papers. It is possible to destroy these enzymes with a heat treatment above 70°C, by catalyst inhibitors, or by a peroxide overdose for a short time.
- Many recovered paper grades contain high amounts of transition metal ions that catalyze peroxide decomposition. Acid pretreatment can remove them or addition of stabilizers can inactive them. Sodium silicate is the most common peroxide stabilizer. It causes problems in the presence of carbonate containing fillers or with use of hard water. An acid treatment is very efficient, but one should restrict its application to recovered paper furnishes with low $CaCO_3$ content. With high $CaCO_3$, high charges of sulfuric acid are necessary. This considerably reduces the filler content of the recovered paper. Another disadvantage of an acid pretreatment is the necessary separation of the acid

and the alkaline water loop leading to higher investment and running costs.

- (OP) treatments are more efficient than common P stages. Often there is only a slight enhancement of brightness but significantly better optical homogeneity. It is possible to reduce more efficiently the mottled appearance of mixed recovered papers—ONP/OMG and MOW—containing brown fibers.
- One can brighten recovered paper furnishes with high mechanical fiber content to 75% ISO in a three stage (OP) P FAS sequence.
- Ozone is a powerful brightening agent for recovered paper furnishes with no mechanical pulp or low content of mechanical pulp. It destroys optical brighteners to a large extent. Ozone can also improve the bonding ability of recovered paper grades having a high content of mechanical fibers. Here one must accept some brightness reduction.
- It is possible to bleach recovered paper furnishes with a low content of mechanical pulp in two stages to brightness values of 80% or even over 85% ISO depending on the raw material, the bleaching sequence, and the chemical charges applied. TJ

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