Peracetate/singlet oxygen chemistry used in post-bleaching of kraft pulp as a practical oxidant for paper machines

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ABSTRACT: The use of a novel sodium peracetate/singlet oxygen chemistry for brightening bleached kraft pulp shows exciting potential for technical performance, supply logistics, safety, and cost reduction. Potential chemical carryover to the paper machine raises questions about whether peracetate will impact paper machine performance, such as metal corrosion, useful press felt life, and interference with existing biocide programs or paper machine chemistry.

Sodium peracetate/singlet oxygen chemistry can be used in high-density storage chests for brightening/whitening and to increase color stability. Any oxidant used directly before the paper machine has the possibility of impacting paper machine operations. Traditional oxidants used in bleaching, such as chlorine dioxide and hydrogen peroxide, are known to cause corrosion on machinery metals and press felts. Hydrogen peroxide residuals can interfere with common biocide programs. Traditional oxidants used in biocide treatments themselves significantly degrade press felt life when the rule-of-thumb concentration thresholds are exceeded. Sodium peracetate is evaluated in this paper for its impact on nylon press felt fiber degradation, metal corrosion, and interference with typical biocide programs.

Laboratory results indicate that sodium peracetate/singlet oxygen chemistry is less corrosive than chlorine, bromine, and hydrogen peroxide on press felt nylon fiber and can therefore be used at higher levels than those chemistries to increase brightness without increasing negative downstream impact. Sodium peracetate can also be used with current biocide programs without negative impacts such as consumptive degradation. Higher residuals of peracetate going to the paper machine may be useful as a biocide itself and can complement existing programs, allowing those programs to stay within their safe operating levels and thereby extend press felt useful life.

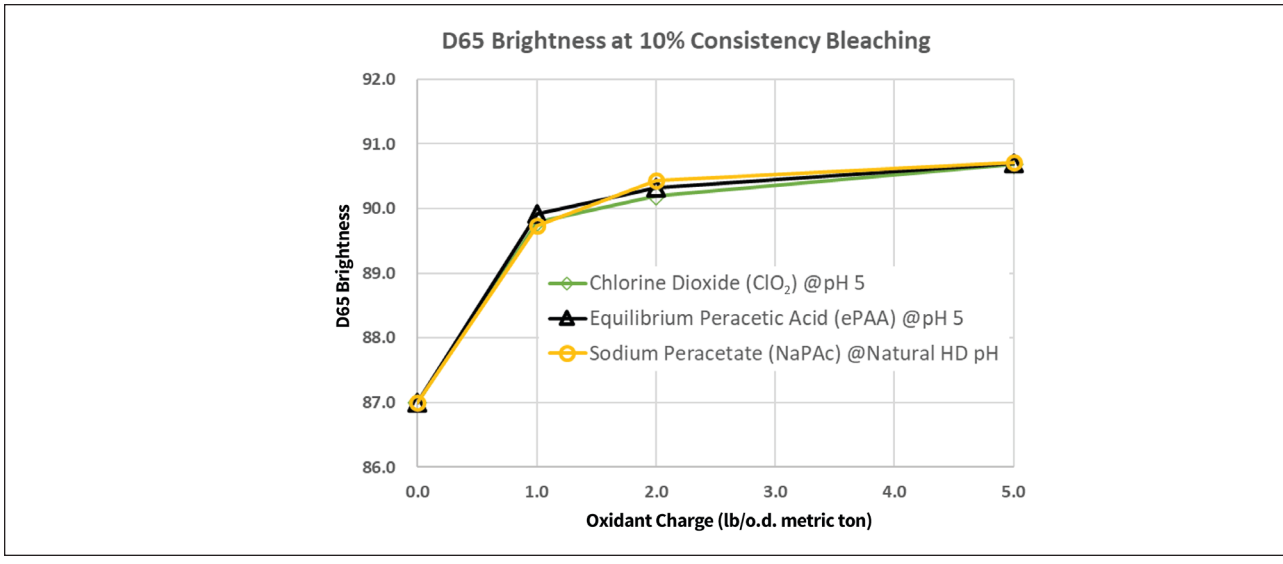
Application: This article introduces readers to sodium peracetate/singlet oxygen chemistry, which is a novel, sustainable, oxidant that is new to the pulp and paper industry and can substitute, or complement, oxidants already in mills. The article covers sodium peracetate use in the pulp mill for brightening pulp and follows through to examine implications of potential oxidant carryover on the paper machine.

The use of peracetate/singlet oxygen chemistry for a variety of applications in pulp and paper production shows unique potential for technical performance, supply logistics, safety, and cost. Recently developed technology to provide singlet oxygen chemistry in a liquid peracetate concentrate has made a new oxidant available that may substitute for, or complement, traditional oxidants like chlorine dioxide (ClO_2), peracetic acid (PAA), or hydrogen peroxide (H_2O_2) in typical pulp and papermaking processes.

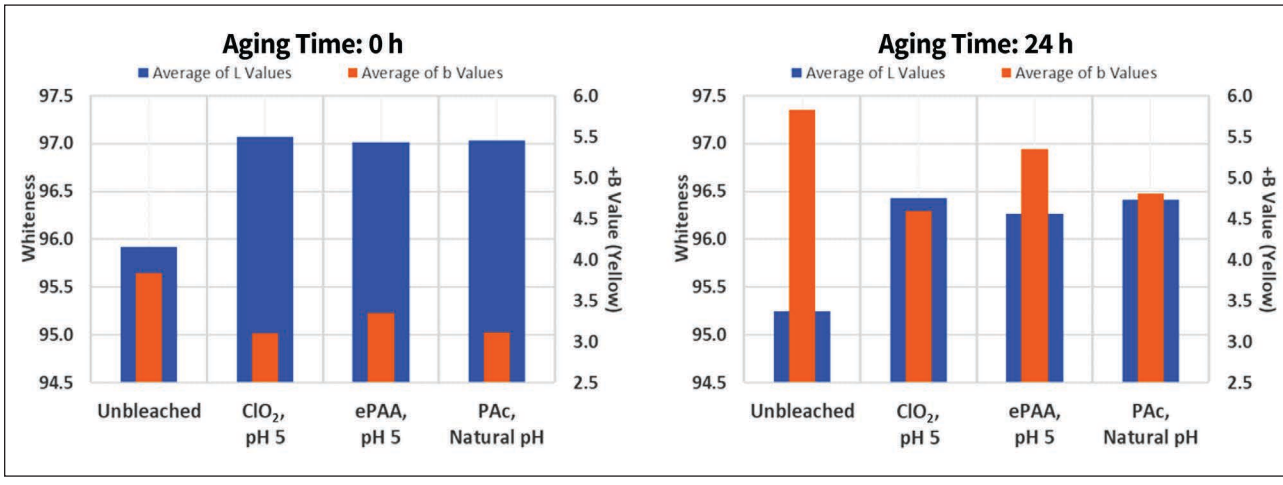
Recent experimental work has indicated that sodium peracetate (NaPAc) exhibits many performance similarities to PAA [1]. Like PAA, which has been extensively studied and used in pulp and paper mills [2-4], the singlet oxygen chemistry has the potential to provide post-bleach brightening/whitening and increase color stability. Sodium peracetate can be easily added in existing high-density (HD) storage chests or in the stock preparation chests. Small charges such as 1–2 lb/metric ton pulp can increase 88% ISO brightness kraft pulp by 1.5 to 2.0 points (**Fig. 1**). It produces whiter pulp with less yellowing (lower *b* value) and, importantly, improves brightness stability by reducing brightness reversion (**Fig. 2** and **Fig. 3**). Decreased reversion can thereby reduce the need for overbleaching to guarantee target specifications.

The use of molecular oxygen in its "singlet" excited electronic state has several attractive qualities as a pulp brightening and/or delignification agent. The electronic configuration of singlet oxygen allows it to undergo a variety of reactions, including formation of endoperoxides, formation of allyl peroxides by Alder-ene reactions, formation of 1,2-dioxetanes, reactions with phenols, reactions with sulfides, and reactions with primary and secondary amines [5,6]. Singlet oxygen is highly reactive toward lignin and other electron-rich materials while being virtually unreactive toward cellulosic fiber.

A patented chemical technology recently developed by Clean Chemistry Inc. (Boulder, CO, USA) generates a liquid



1. Brightness gain curves from various oxidants at 10% consistency.

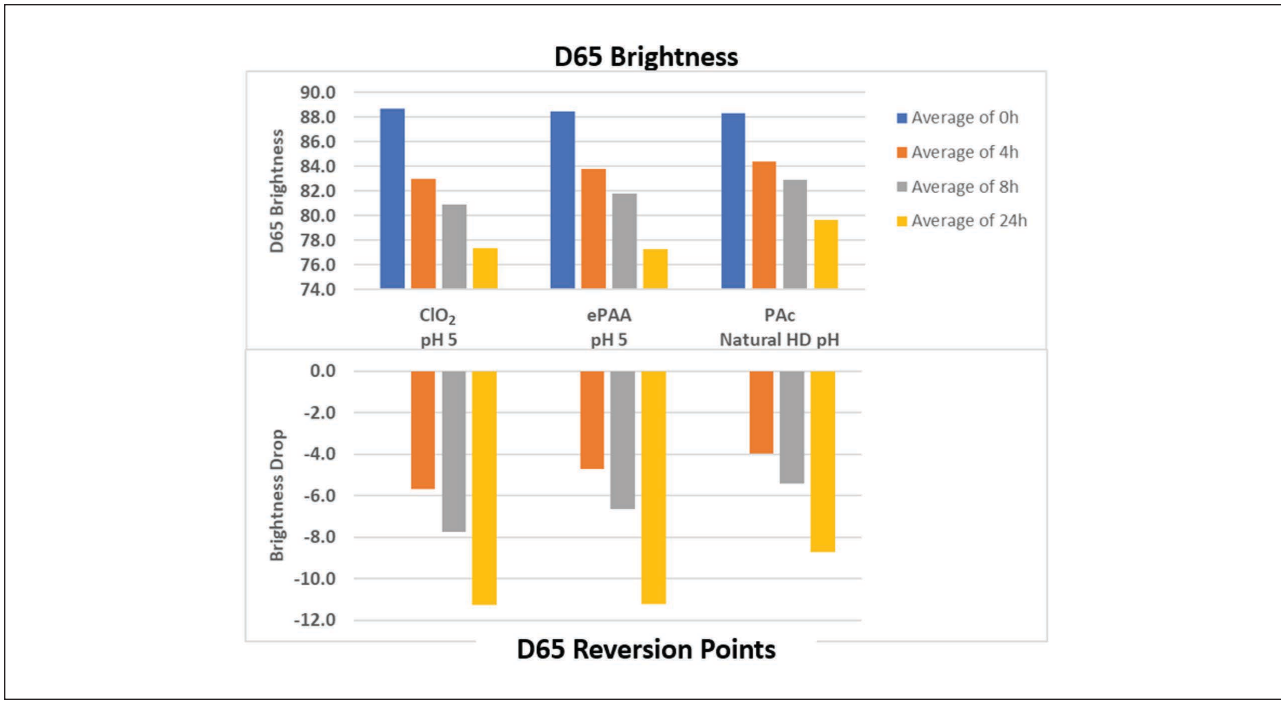


2. Whiteness and yellowing comparison for pulp brightened with 5 lb/o.d. metric ton oxidant charge at 0 h (left side) and 24 h (right side). Untreated control samples were included for comparison.

"parent" oxidant solution specially formulated to efficiently produce singlet oxygen in-situ by the decomposition of NaPAc. The product is free of chlorides and sulfates, and the byproducts are glycerin and sodium acetate. The technology is inherently safer, less corrosive, and has very low adsorbable organic halides (AOX) formation potential relative to chlorine and ClO_2 [7]. Earlier results show a high level of activity and selectivity for reactions of singlet oxygen with lignin (i.e., kappa reduction), as well as brightening of hardwood and softwood pulps. This technology has been successfully scaled-up for industrial applications and has been used for oilfield water treatment and reuse since 2014.

The equipment used for generating and dispensing the sodium peracetate product is a low power, low pressure, automated system of metering pumps and an inline reactor process contained in a relatively small footprint. The capital and operating costs of such equipment is minimal compared to that of a ClO_2 plant, distilled PAA plant, or even PAA storage facilities.

Improving the brightness and color stability of kraft pulp is a desired outcome for many papermakers; however, the use of oxidants directly before or on the paper machine has the possibility of negatively impacting paper machine operations. It is known that traditional bleaching oxidants, such as chlorine dioxide and hydrogen peroxide, can cause corrosion on machinery metals and seriously damage paper machine clothing, such as nylon press felts. It is also true that other oxidants, such as residual hydrogen peroxide, can interfere with common biocide programs by neutralizing chlorine- and bromine-containing compounds. Also, oxidants used directly on the paper machine as biocide treatments significantly degrade press felt life when best practice, safe-harbor concentration levels are exceeded. Published laboratory studies have correlated fabric resistance to wear in abrasion tests with intrinsic viscosity and



3. Reversion test results for pulp brightened with 1 lb/o.d. metric ton oxidant charge. Measured D65 brightness over time (upper panel) and D65 reversion from initial, 0 h (lower panel).

fiber loss accelerated by exposure to hypochlorite and hypobromite solutions [8]. Similar work has investigated the impact of hydrogen peroxide and chlorine dioxide on paper machine felts at concentrations observed in carryover from the bleach plant [9].

To establish the suitability of NaPAc as a practical oxidant for paper machine compatibility, this paper compares various oxidants for their impact on press felt nylon degradation. It also covers topics of chemical compatibility of oxidants with biocides and metal corrosion.

METHODS

A method was developed to study oxidant exposure to the nylon fibers that provides strength and functionality to press felts. New 11-dtex polyamide (PA) 6.6 nylon press felt batting fiber (Albany International; Rochester, NH, USA) was immersed and mixed in a 1 g/L solution of Triton X-100 surfactant in distilled water overnight to remove surface treatments during manufacture. The fiber was rinsed thoroughly with distilled water and air dried for at least 12 h. Washed fiber was cut into 2.7 g "coupons" that were preweighed and put into polypropylene mesh holders attached to the end of stainless-steel mixer shafts. The shaft-mounted coupons were installed onto variable speed overhead mixers with the coupons immersed in temperature-controlled baths containing the test oxidant solutions. The coupons were rotated at about 60–80 rpm in the solutions. Total exposure time was 200 h at 50°C. "Control" samples of fiber (no oxidant exposure) were immersed in dechlorinated tap water held at the same temperature for the test period.

Exposure bath makeup solutions of common oxidants (sodium hypochlorite, sodium hypobromite, and hydrogen peroxide) in dechlorinated tap water were made fresh at least twice per day and their concentrations were verified. Sodium hypochlorite was used as the source of aqueous chlorine. Water chlorinated with sodium hypochlorite contains a pH-dependent composition of three species: molecular chlorine (Cl_2), hypochlorous acid (HOCl), and hypochlorite (OCl^-) [10]. The HOCl/OCl^- species are in an acid-base equilibrium with one another (pKa ≈ 7.5 at 25°C) and typically comprise the majority of chlorine species in solution above about pH 3. For simplicity, measured quantities of total chlorine are referred to in this text as "chlorine." Similarly, sodium hypobromite was used as aqueous bromine. Sodium hypobromite solutions contain a pH-dependent composition of molecular bromine (Br_2), hypobromous acid (HOBr), and hypobromite (OBr^-). The HOBr/OBr^- equilibrium (pKa ≈ 8.7 at 25°C) favors the more active HOBr form at higher pH as compared to the HOCl/OCl^- equilibrium. Measured quantities of total bromine are referred to in this text as "bromine."

Makeup solution pH adjustments were made with 4 N sulfuric acid or 1 N sodium hydroxide solutions to maintain a target pH of between 7.5 and 8.0, similar to a fine paper machine. Makeup solutions of hypochlorite, hypobromite, and hydrogen peroxide were fed with peristaltic metering pumps into the exposure baths every 30 min, displacing 12.5% of the _volume. Sodium peracetate solution was produced continuously from a laboratory-scale production system that included a metered dechlorinated tap water feed

for dilution of the product solution prior to metering continuously into the exposure bath. Makeup solution concentrations (or peracetate metering rate) were adjusted to maintain the target oxidant concentration in the exposure baths.

Exposure bath conditions were measured several times per day for pH, temperature, and oxidant concentration. Total chlorine was measured by the *N*,*N*-diethyl-*p*-phenylenediamine (DPD) colorimetric method (HACH Method 10070, HACH; Loveland, CO, USA). Total bromine was measured by a similar DPD method (HACH Method 8016). Hydrogen peroxide concentration was measured by iodometric titration (potassium iodide [KI], starch, ammonium molybdate/sulfuric acid [H_2SO_4], and sodium thiosulfate titrant). Sodium peracetate concentration was measured by standard iodometric titration (note that peracetate solution contains no hydrogen peroxide to interfere).

Upon conclusion of the exposure test period, the coupons were removed from the exposure baths, rinsed thoroughly with distilled water, soaked for about 2 min in 0.01 N thiosulfate solution, and thoroughly rinsed with distilled water. The felt fiber samples were removed from the polypropylene mesh holders and air dried for at least 12 h. Final dry weights of each coupon were measured to determine weight loss.

Felt fiber coupons were evaluated by microscope imaging, intrinsic viscosity, and tensile strength. Bright field optical microscope images were made using a Keyence VHX-1000 digital microscope (Keyence; Osaka, Japan) with a VH-Z100 lens magnification of 100X–1000X. Scanning electron microscope (SEM) images were obtained on samples sputter coated with gold and imaged using a JEOL JCM-6000 Plus Benchtop SEM (JEOL; Tokyo, Japan). Intrinsic viscosity of felt fiber material was measured using an Anton Paar IV Microviscometer (Lovis 2000 M model, Anton Paar; Graz, Austria) and calculated with the Billmeyer equation. Tensile strength of the 11-dtex fibers was measured on an EMIC/Instron Dynamometer (DL-2000 model, Instron; Norwood, MA, USA).

RESULTS

The primary performance comparisons were made among the oxidants at two different levels of charge (**Table I**). The lower level of charge, designated as the "safe harbor" charge level, is based on various lab studies and mill-based experiences, and is documented in several publications [8,9,11]. It is generally believed that if the oxidant concentration is kept below this level, then the chemical degradation of the press felt does not materially add to the shortening of the press felt life. This, however, does not mean that there is no chemical damage, only that it is not the major contributor to the felt's failure.

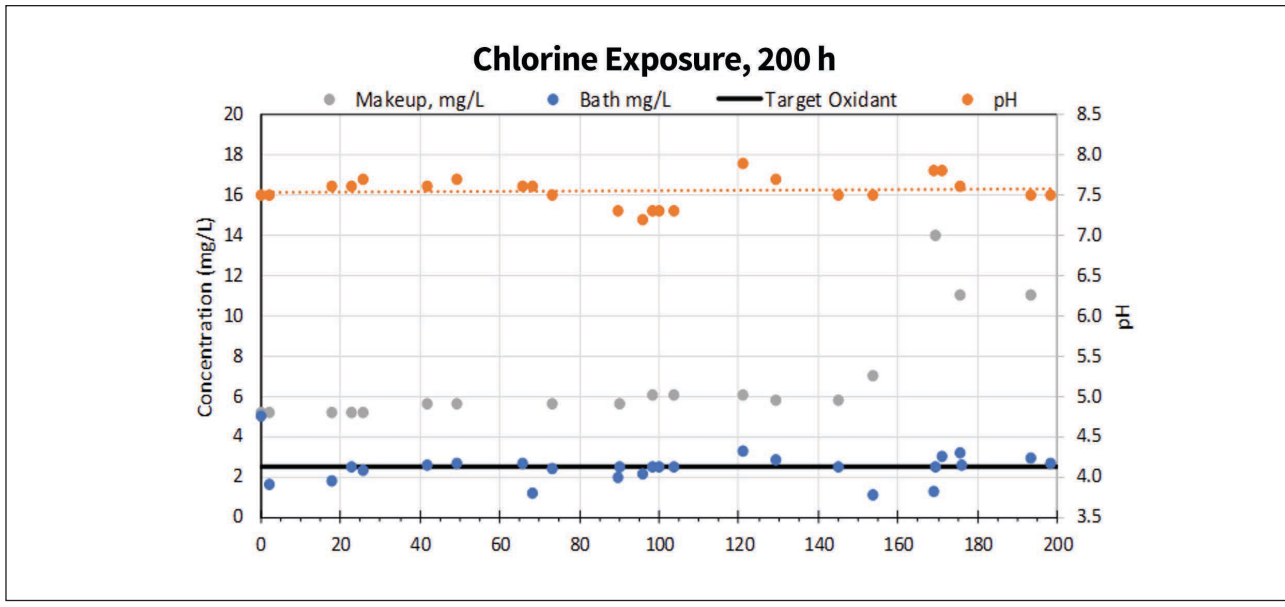
Due to lack of field experience with the newly invented sodium peracetate, there is not an established "safe harbor" level, so the lower limit was chosen based on a useful biocide efficacy level. Assuming each oxidant must be able to control microbes at its lowest acceptable level of use, then using a typical dose for a biocide application is appropriate. Sodium peracetate residuals of from 2 to 5 ppm are typical when using sodium peracetate as a biocide. The upper end of this range was chosen to be conservative. The higher charge level for all oxidants was chosen as five times the safe harbor level for the purpose of clearly identifying chemically induced damage.

	Low Charge	High Charge
	"Safe Harbor," ppm	5 x "Safe Harbor," ppm
Chlorine	0.5	2.5
Bromine	0.3	1.5
Hydrogen peroxide	100	500
Sodium peracetate	5	25

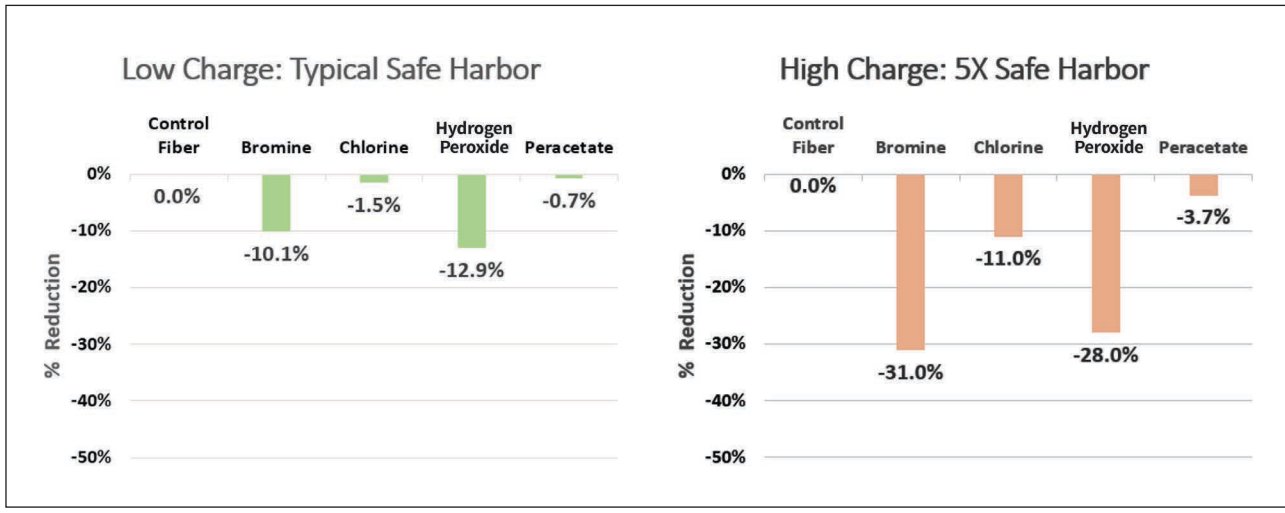
I. Chemical bath concentration levels for degradation testing of press felt nylon fiber.

Maintaining the target concentration of the oxidant in the water bath required frequent monitoring. **Figure 4** shows the target chlorine oxidant level in the bath for the high charge experiment. The oxidant is continually consumed and must be added at regular intervals at a significantly higher level to maintain the target. It is interesting to note that after a certain amount of exp+osure time, the rate of oxidant consumption increases as the fiber starts to corrode more rapidly. This was evident from distinct increases in makeup solution concentrations needed to maintain the target bath concentration. During the high charge experiments, these increasing trends were observed for chlorine (after 150 h), bromine (after 40 h), and hydrogen peroxide (after 120 h). No increase in consumption rate was detected for the sodium peracetate chemistry. Similar consumption trends were observed for chlorine and bromine at the lower, "safe harbor," concentrations. It appears that beyond a critical damage threshold, the oxidant consumption rate (and fiber corrosion rate) can increase as the reactive surface area of the fiber increases at a microscopic level and/or the stability of the nylon material itself decreases by damage at the molecular level. For example, the bromine-exposed fiber samples (and chlorine to a lesser extent) appeared pale green after 1–2 days of exposure at the high charge level, indicating a chemical change in nylon polymer structure. Whatever the causes may be, the net result is loss of felt fiber mass and integrity.

The most immediate indication of corrosion to the fiber is based on mass loss (**Fig. 5**). Simple weighing of the treated sample and comparing it to the new fiber indicates how much material is removed by the oxidant. At both levels of charge, it is evident that bromine and hydrogen peroxide most aggressively degrade the nylon fiber. At low



4. Managing and monitoring oxidant concentration and pH at 50°C—chlorine example.

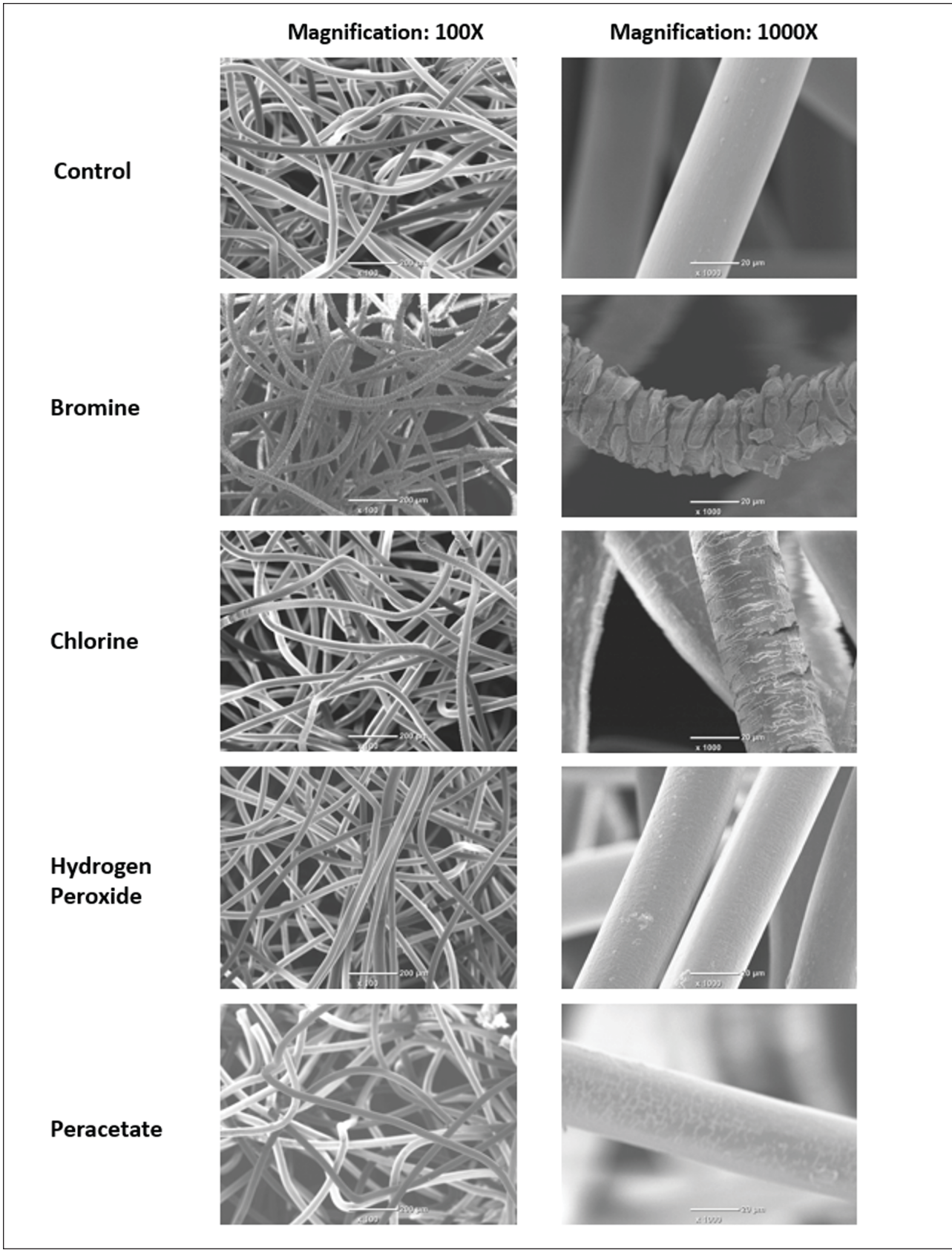


5. Nylon fiber mass loss.

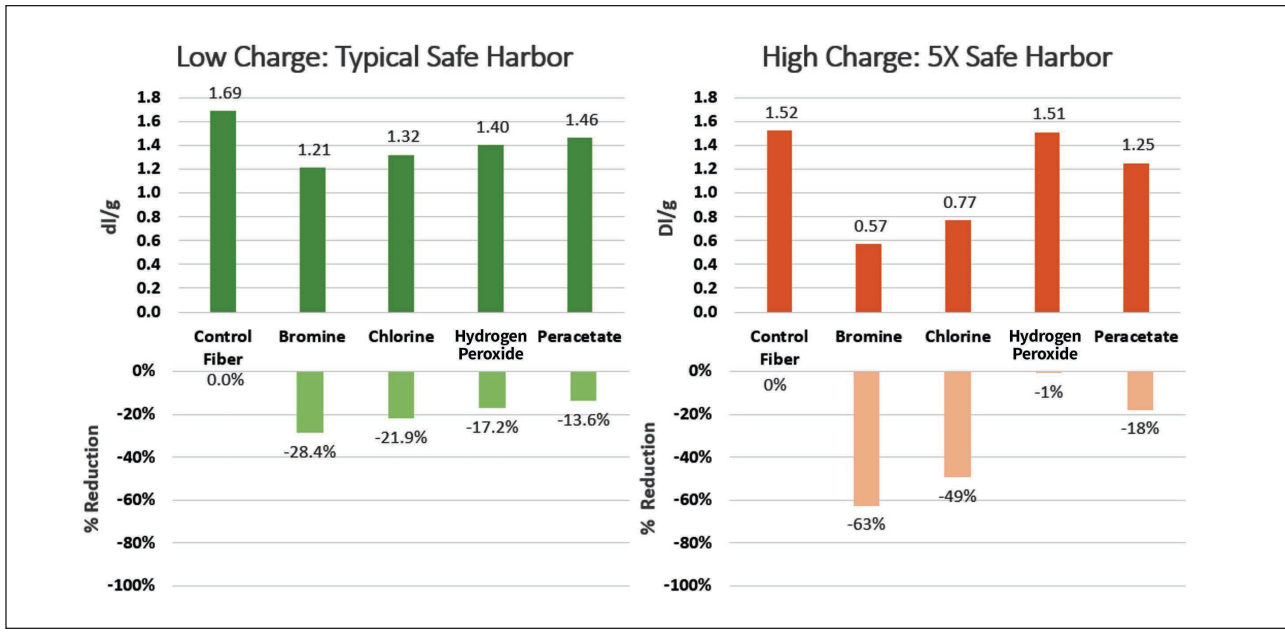
dosage, very little material is removed from the chlorine sample, which is consistent with the safe harbor dosage widely used. However, as the dosage is increased, chlorine removes a substantially higher amount of material in proportion to the increase in charge. Sodium peracetate removes the least amount of material in both scenarios.

How the nylon material is removed can vary greatly based on the mechanism of the oxidant (**Fig. 6**). For example, bromine and hydrogen peroxide both remove a similar amount of mass from the nylon. Looking at SEM images at 1000X magnification provides insight into how the nylon is attacked and possibly how the remaining nylon will behave in further testing. Hydrogen peroxide appears to remove the nylon almost evenly from the outer surface, with very little pitting into the nylon. Bromine severely pits the surface and creates crevices in the core of the fiber. While the control fiber remained unchanged in mass and appearance, all other oxidants caused mass loss consistent with some type of physical damage.

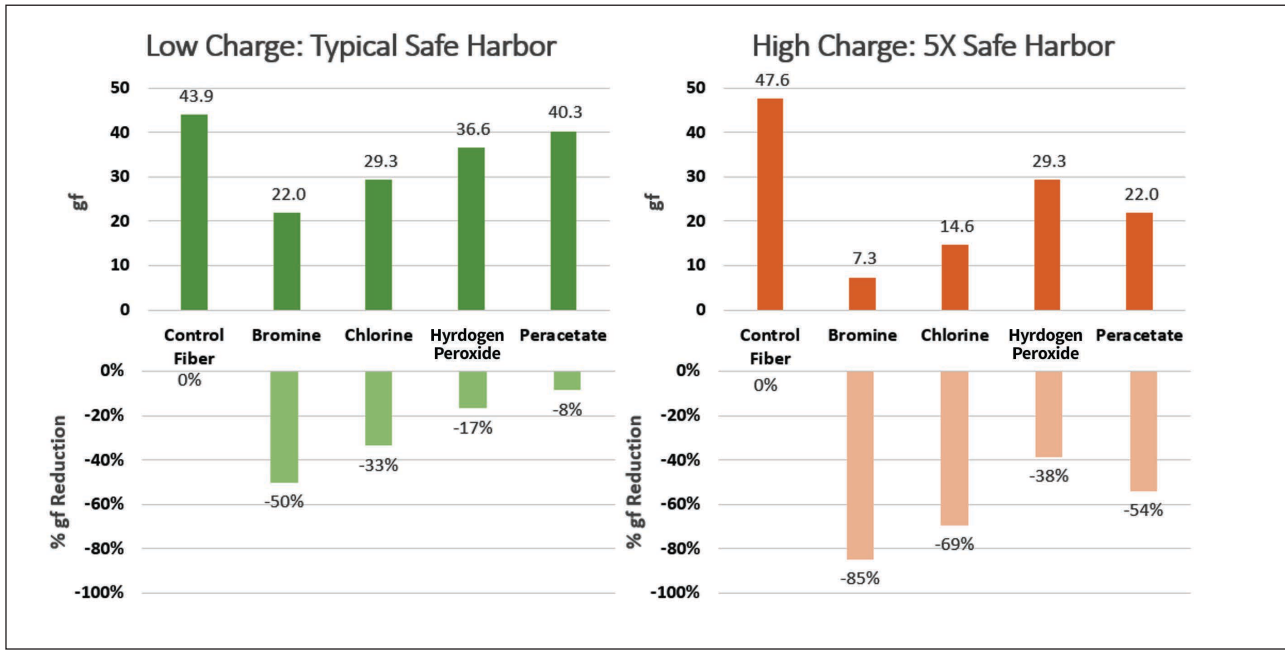
A common method for evaluating nylon destruction is the measurement of intrinsic viscosity (**Fig. 7**). At the low charge of oxidant, we can see a clear order of how oxidants attack the nylon, with bromine being the most severe and sodium peracetate being the least severe. The intrinsic viscosity measurement is not materially impacted at the high-level hydrogen peroxide charge. This may be due to how the hydrogen peroxide removed the nylon. If surface nylon is removed but the core remains relatively intact, then the integrity of the remaining nylon fiber may be undamaged. In essence, the intrinsic viscosity of the material removed cannot be measured and only the material left is evaluated. This hypothesis can be further supported by comparing



6. Scanning electron microscope (SEM) comparison of chemical degradation at 100X and 1000x magnifications for high charge treated samples.



7. Nylon fiber intrinsic viscosity loss.



8. Nylon fiber tensile strength loss.

the treated fiber's measured diameter to that of the control fiber. The hydrogen peroxide fiber diameter decreased by 16.2% and the cross-sectional area decreased by 29.8%. Since intrinsic viscosity testing is based on mass, the sample material tested contained more undamaged material from the core of the nylon fiber.

Viscosity measured for the sodium peracetate high charge sample is similar to the viscosity of the other oxidants in the low charge samples. Combining this observation with a relatively low loss of mass for the sodium peracetate indicates that this chemistry does not materially remove the nylon fiber as compared with the other oxidants.

Tensile strength testing of the treated samples follows the same pattern as the intrinsic viscosity results (**Fig. 8**). Hydrogen peroxide activity and damage mechanisms appeared to change with concentration, presumably due to several factors that impact its chemical activity and reactive decomposition products, including pH and trace metals.

Summarizing the nylon press felt oxidant study, prolonged exposure to each oxidant has negative impacts on nylon properties, even at low, normally acceptable, levels of charge. Sodium peracetate was found to be the least de-

Parameter	Untreated	5 ppm NaOCl	5 ppm NaOCl + 13 ppm NaPAc	Comment
pH	7.28	7.39	7.56	No material pH change
ORP (mV vs. SHE)	474	652	708	Increased ORP
ATP, pg/mL	4790	249	51.7	Substantial ATP decrease
% ATP reduction	0	94.8%	98.9%	
Free Cl_2, mg/L	0	<0.5	<0.5	No change to Cl levels
Total Cl_2, mg/L	0	3	3	No change to Cl levels

NaOCL = sodium hypchlorite; NaPAc = sodium peracetate; ORP = oxidation-reduction potential; SHE = standard hydrogen electrode; ATP = adenosine triphosphate; Cl_2 = chlorine.

II. Sodium peracetate (NaPAc) is compatible with hypochlorite and enhances biocide activity.

structive at safe harbor levels. At these levels of oxidant exposure, mechanical wear may become the dominant cause of failure. High levels of oxidant usage materially degrade the nylon strength and can become the key driver of failure in press felts. When excessive oxidant exposure is combined with mechanical stress, press felts are known to fail much more quickly, with some mills experiencing 75% reduction in felt life. Sodium peracetate is generally less corrosive than the other oxidants and, even at five times the charge of the other oxidants, results in less mass and intrinsic viscosity loss.

When used for HD tower brightening, the likelihood of high levels of sodium peracetate carryover is exceedingly low, as one of the fundamental attributes of the chemistry is its short lifespan after manufacture. Residual concentrations of sodium peracetate after normal brightening usage (approximately 1 to 2 lb/metric ton) and lengthy storage time in the HD chest (from 4 to 12 h) will consistently approach zero ppm.

Another concern of introducing an oxidant onto the paper machine is how it will behave with the current biocide program. Typical biocide programs that are chlorine- or bromine-based are easily disturbed with carryover hydrogen peroxide. In fact, hydrogen peroxide is used commercially to dechlorinate water [12].

Sodium peracetate/singlet oxygen chemistry is compatible with chlorine (and bromine) products used in water treatment applications. **Table II** shows the impact of combining the two oxidants that reinforce one another to provide additive performance benefits for microbial control [13]. In this example, a recycled oilfield water source for use in well completion operations was heavily contaminated with bacteria, fungi, and algae. Water samples were treated with a one-time addition of sodium hypochlorite alone or a combination of sodium hypochlorite and sodium peracetate to demonstrate the impact of their combined use on

Corrosion Rates in mpy	O_2 8-9 ppm	NaPAc 13 ppm
Carbon Steels		
C1018	27.7	33.1
Ductile	34.0	34.5
Pipe L80	37.92	41.2[a]
Stainless Steels		
CA6NM	0.97	0.62
17-4PH	0.00	0.29
304L	0.08	0.03
CD4MCUN	0.10	0.01

[a] 120 ppm sodium peracetate (NaPAc).

III. Sodium peracetate (NaPAc) has similar corrosivity to dissolved oxygen for materials commonly found in paper mills.

treatment rate and total chlorine residual. Total microbial activity was evaluated by measuring adenosine triphosphate (ATP) using the Luminultra ATP test method. The ATP level was reduced by about 95% after 60 min of treatment time with sodium hypochlorite, while ATP was reduced by about 99% after 60 min of treatment time with the combined oxidants. Total chlorine residual was similar for sodium hypochlorite and the combined oxidant tests. The oxidation-reduction potential (ORP) of the treated water was highest for the combined oxidant treatment, which also reflects an additive benefit rather than consumptive interference. Carryover of the sodium peracetate/singlet oxygen chemistry onto the paper machine is expected to be a supplementary enhancement to existing biocide programs.

Paper machine equipment, including felt rolls, dryers, pumps, etc., are susceptible to corrosion from carryover of conventional oxidants or excessive levels of oxidizing biocides [14]. While the exact conditions of corrosion and resulting corrosion rates are highly variable throughout a paper machine, a relative comparison can be made to benchmark the sodium peracetate/singlet oxygen chemistry. It has been demonstrated in laboratory testing and field experience that the corrosive behavior (rates and uniformity) of the sodium peracetate/singlet oxygen chemistry is very similar to dissolved oxygen under aggressive conditions [15]. **Table III** compares the corrosion rates for a variety of metallurgies encountered in process equipment exposed to dissolved oxygen and sodium peracetate solutions. The "short-term" corrosion coupon tests (following ASTM G4 guidelines) employed a standard corrosion coupon rack in a flow loop configuration at room temperature. Oxidants were maintained at the target concentrations in dechlorinated tap water near pH 8 containing 1% sodium chloride, which was added to simulate a more aggressive dissolved solids composition. A general "rule-of-thumb" is that every 13 ppm of sodium peracetate has the potential to release 2 ppm of dissolved oxygen if it is not otherwise consumed as it naturally attenuates. The end result is that a modest carryover of sodium peracetate onto the paper machine will be less corrosive than comparable concentrations of chlorine, bromine, and chlorine dioxide, which are significantly more aggressive than dissolved oxygen, especially at elevated temperatures encountered on paper machines.

CONCLUSIONS

Lab studies indicate that sodium peracetate/singlet oxygen chemistry is a viable chemistry for post-bleach brightening of kraft pulp in an HD chest. Sodium peracetate application of 1–2 lb/metric ton of kraft pulp can deliver about 2 points of brightness gain, which is more resistant to reversion than when bleaching with other brightening agents. The experiments contained in this paper indicate that sodium peracetate will not cause practical problems if small amounts of oxidant carry over to the paper machine—a common concern for traditional oxidants. Sodium peracetate is shown to be less corrosive on press felt nylon than other traditional pulp and paper oxidants and is minimally damaging at safe harbor charge levels. Also, sodium peracetate will not interfere with traditional biocides used on paper machines and may, in fact, boost the effectiveness of chlorine-based biocides. Sodium peracetate is also less corrosive on common paper mill metals than equal amounts of the other oxidants. In total, this work should assuage concerns that carryover of sodium peracetate as a bleaching agent will cause unanticipated problems on the paper machine.

Sodium peracetate is a practical oxidant for paper machines and can be safely used in HD tanks at existing conditions. It is a drop-in chemistry that requires no capital

ABOUT THE AUTHORS

This work was an opportunity to investigate a potentially useful application of the relatively new peracetate/singlet oxygen chemistry, which exhibits unique capabilities and safety compared to conventional oxidants. I (Buschmann) have previously investigated and published results for using this chemistry for pulp delignification, brightening, microbial control, and pollution reduction. The current work investigates important considerations for this chemistry's use that may impact the papermaking process and equipment.

Determining how to construct laboratory protocols to evaluate corrosion processes on the paper machine press felts was challenging, but through the help of prior articles and industry experts, we created a robust experimental process.

Research on this novel oxidant chemistry with nylon fabrics had not been previously examined, and this work has provided the basis for understanding how the peracetate/singlet oxygen chemistry may be beneficial and preferable to other oxidants for cleaning nylon and textiles and for other industrial applications. The most surprising outcome of the work was to discover the wide variety of forms that corrosion takes based on each oxidant chemistry used under each specific condition.

A major benefit for mills from this study is introduction of a novel oxidant that can give mills increased flexibility in designing processes for improved sustainability and reduced cost. There are many applications to explore for peracetate/singlet oxygen, including delignification, brightening, microbial control, odor control, water treatment, and other uses where it can replace or complement common oxidants and other products used in pulp and paper mills and where a low-capital, sustainable solution would be preferred.

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investment so it can be deployed quickly vs. other brightening chemistries for mills that require brightness enhancement. **TJ**

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