

A PILOT PLANT TRIAL USING *IN SITU* PEROXYBORATE FOR THE BRIGHTNESS RETENTION OF TMP

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ABSTRACT

The possibility of using peroxyborates, produced *in situ* by combining aqueous alkaline hydrogen peroxide and borax, for bleaching and brightness reversion inhibition of thermomechanical pulp was investigated. The peroxyborate material was applied to BTMP paper produced in the laboratory, and from a pilot scale paper making trial. Excellent brightness retention characteristics were exhibited in both the laboratory and pilot scale paper making *in situ* peroxyborate application trials. A number of mechanical and structural properties of the peroxyborate paper showed significant improvement compared to control samples, or samples with hydrogen peroxide or borax applied alone.

Application:

This peroxyborate application may be useful for mills experiencing brightness loss during paper storage.

Environmental concerns and government regulations are continuously forcing the pulp and paper industry to adopt new technologies to reduce the impact that pulping and bleaching plant effluent has on receiving waters and the atmosphere (1). In particular, chemical compounds that contain chlorine, generally referred to as chlorinated organics, have been identified as the major source of concern. The need to produce high quality, high brightness pulp with minimal environmental impact is being faced by mills world wide (2).

As well as the environmental concerns, economic factors play a very important role in the type of pulping and bleaching processes that a mill may choose to utilize. Newsprint, advertising inserts, directory paper and some catalogue paper are considered short-life products. They are produced relatively cheaply from lignin-containing (high yield) thermomechanical pulp (TMP) and chemithermomechanical pulp (CTMP). High yield pulps make effective use of wood resource and have attractive bulk, absorbency, optical, and printing properties (3). Increasing wood costs and predicted shortages in future wood supplies continue to stimulate interest in high yield pulp production and use (4).

However the relatively low brightness of the high yield pulps is the most serious limitation for their use; as well, paper produced from this grade of pulp turns yellow very rapidly upon exposure to heat or light, limiting its life span. It has been suggested that if the time taken for the light induced yellowing of these papers could be increased 3-36 months, the potential market for bleached TMP (BTMP) and CTMP would be expanded by 0.6-2.2 million tons per year (5). If the tendency to yellow could be prevented, BTMP and CTMP could be included (in

greater proportions) to furnishes used in the manufacture of high brightness papers. Oxidative, thermal discoloration through long storage time at ambient temperature can result in brightness losses of 2-5 points, and increase with higher temperatures and humidity (6). Although much research has been devoted to finding better methods of bleaching these types of pulps, and for improving their brightness stability, the results so far do not look very promising.

Hydrogen peroxide (H_2O_2) is a very versatile reagent used in wood pulp bleaching because of decreased environmental emissions, lower bleaching costs, and improved pulp quality (7). However, the high brightness levels of paper that consumers desire cannot be achieved for high yield pulps by using hydrogen peroxide alone; high yield pulps bleached with hydrogen peroxide alone still tend to revert to a yellow color in a relatively short time. For this reason, research investigating the transformation of hydrogen peroxide into other more reactive peroxygen compounds has been one of the approaches used to increase the effectiveness of peroxide bleaching (8). The paper will explore the use of one such option- the addition of borax (sodium tetraborate decahydrate) to aqueous alkaline hydrogen peroxide- to produce *in situ*, active oxygen peroxyborate species which may increase the bleaching ability allowing for improvement in the initial brightness and long term brightness stability of thermomechanical pulps. The peroxyborate material will be applied after paper formation because it will eventually be tested for synergistic improvements in brightness retention when applied with a sprayed-on ultraviolet (UV) screen.

BACKGROUND

Borax (sodium tetraborate decahydrate) is an important polyborate compound, in terms of both tonnage and dollar sales. It has traditionally been used as a household cleaner and laundry agent; as environmentalists have grown increasingly concerned about the use of phosphates in laundry detergent, some consumers have returned to the use of borax cleaners.

In aqueous alkaline solution, borax forms an extensive pH dependent equilibrium system with other borate compounds. In dilute solution depolymerization occurs rapidly, thus the mononuclear species borate ($B(OH)_4^-$), along with H_2O_2 , is formed when borax is dissolved in aqueous solution. Increasing hydrogen peroxide (H_2O_2) concentrations and/or increasing pH lead to monomeric peroxyborates (peroxoborates) (9). There have been several studies of the interaction of borate anions with hydrogen peroxide in aqueous solutions (10). The equilibrium formed is fairly rapid; free H_2O_2 or its anion will predominate in dilute aqueous systems, and thus be responsible for most of the oxidizing activity, but there are indications that the peroxyborate species add to the reactivity as functional group oxidants, or bleaching agents. One explanation is that this equilibrium has the ability to deliver the perhydroxyl anion (HO_2^-) at a lower pH than is usually available (pKa for H_2O_2 is ~11.6), giving an advantage in nucleophilic oxidations (10).

It has been suggested that the brightness ceiling of mechanical pulps may be due to limited access, in conventional bleaching processes, of the hydrogen peroxide to the chromophores. It has been determined that about 60% of coniferaldehydes can be removed during peroxide bleaching, but complete removal of the coniferaldehydes can be achieved when isolated lignin is treated with the peroxide bleaching agent (11). These results imply that a higher pulp brightness value could be reached, using a peroxygen compound, if accessibility to the chromophores could be improved. This bleaching improvement may be realized with the addition of peroxyborate species.

Borates are known to form esters with hydroxy compounds in aqueous environments (12). The vicinal diols present in carbohydrates can form a number of reversible ternary complexes with hydrogen peroxide and borate in alkaline aqueous environments (13,14). It is believed that it is this relative mobility of the peroxyborate functionality which leads to an increased bleaching capability and a longer retention of the bleaching components (15). It has been hypothesized that once a perborate ester has oxidized a chromophore at one location, the reversibility of the peroxyborate-carbohydrate complex formation would allow for migration of the peroxyborate species to a new site in the ligno-cellulosic complex (14). Regeneration of the perborate group through reaction with free hydrogen peroxide or perhydroxyl ions in solution, or by oxidizing-reducing coupling reactions within the fibre matrix, permits the interaction with and subsequent oxidative destruction of other chromophoric groups within the lignin. This theory helps to explain the potential advantage of adding peroxyborate compounds during peroxide bleaching of mechanical pulps.

This project was designed to examine the bleaching and brightness reversion inhibition effects of peroxyborate compounds produced by combining aqueous alkaline hydrogen peroxide and sodium tetraborate decahydrate (borax) at a laboratory scale level. It is believed that this *in situ* production of solid state bleaching compounds provides a synergistic advantage in the bleaching and brightness retention of pulp compared to the action of hydrogen peroxide or borax alone.

The feasibility of applying a combination of aqueous alkaline hydrogen peroxide and borax during BTMP paper production was also to be studied, and it would be determined if this peroxyborate system could be applied to a wet web on a pilot scale paper making machine. A pilot scale trial would allow for a large production of the this paper so that extensive optical, structural, and mechanical tests could be performed. The pilot trial would also identify any problems that might arise during the peroxyborate application procedures, and thus these could be corrected before mill scale work was carried out.

EXPERIMENTAL

BTMP handsheet preparation

Bleached thermomechanical pulp (BTMP) handsheets for laboratory scale experiments were prepared as follows. Black spruce (*Picea mariana*) thermomechanical pulp (TMP) was washed with 0.2% DTPA at 70EC for 30 minutes followed by a cold deionized distilled water rinse. The TMP was bleached using 0.05% MgSO₄, 0.2% DTPA, 3% Na₂SiO₃, 2.5% NaOH and 4% H₂O₂ (all solutions prepared using deionized distilled water) at a 10% pulp consistency. The bleaching was carried out in a sealed polyethylene bag in a 70EC water bath for two hours, after ensuring all air was removed from the bag. After one hour in the water bath, the bag was withdrawn, so that any unremoved air could be pressed out; the bag was then resealed and placed back in the water bath for the second hour. At the end of the two hour period, the sealed bag was removed from the water bath and rinsed under cold running water to quench the bleaching reactions.

Irradiation protocol

The pulp sheet samples were irradiated using 30x30x30 cm photoreactors. The photoreactor units could supply both fluorescent or ultraviolet irradiation under controlled conditions. Handsheet samples were placed on the floor of the box and were irradiated by an eight position light source on the inner surface of the top of the box. A digital light meter was used to measure the illuminance (in foot candles (fc)) on the floor of the photoreactor.

Chemical additive solution preparation

The chemicals used in the laboratory bleaching experiments were reagent grade hydrogen peroxide (Caledon), borax (sodium tetraborate decahydrate) (Aldrich), and combinations of hydrogen peroxide and borax. The pH was adjusted using sodium hydroxide (NaOH) (BDH). Freshly prepared chemical solutions were added on a w/w % basis to both sides of a handsheet. The bulk chemicals used in the pilot trials were hydrogen peroxide (35%, technical grade, Fisher), anhydrous borax glass (technical grade, Anachemia), and sodium hydroxide (pellets, Fisher), and were applied to one side of the wet web.

RESULTS AND DISCUSSION

Laboratory irradiation trials

The initial laboratory scale experiments performed were short term (accelerated aging) trials in which hydrogen peroxide, borax, and combinations of hydrogen peroxide and borax (*in situ* peroxyborates), were applied to BTMP sheets so as to compare the bleaching and brightness reversion characteristics using different chemical applications. Accelerated aging experiments were carried out using the photoreactors with UV or strong fluorescent irradiation (much stronger irradiation than what paper products would normally be exposed) to decrease the length of time of the experiments.

The short term accelerated aging trials demonstrated that an *in situ* application of peroxyborates, even in the presence of strong UV and fluorescent irradiation, allowed for better long term brightness reversion inhibition compared to applications of hydrogen peroxide alone, or to control NaOH applications. The optimal charges for the H₂O₂ and borax were determined to be 3% by weight (o.d.) for both species, applied at a pH of 10.8. These results indicated the importance that pH has on the long term brightness recovery of BTMP samples treated with hydrogen peroxide and borax; higher alkali charges shift the perhydroxyl-hydrogen peroxide equilibrium towards the production of more perhydroxyl ions, considered to be responsible for the main bleaching activity in mechanical pulp peroxide bleaching (16), and increased peroxyborate concentrations (17), although any higher alkali charges could promote increased yellowing due to the alkali darkening effect (18). Based on these results, the following long term (ambient-like irradiation) laboratory experiments were performed, concentrating on the application of (*in situ*) peroxyborates produced by combining aqueous alkaline hydrogen peroxide and borax.

In the first long term irradiation experiment, BTMP handsheets were exposed to direct outdoor spring sunlight for 7 hours, after which time the handsheets were stored in the dark. ISO brightness measurements were recorded so as to follow the progress of solid state (dark) bleaching. The brightness of the handsheet samples increased approximately 1-2 points with the

addition of 3% H₂O₂ or a combination of 3% H₂O₂/3% borax prior to light exposure, indicating initial bleaching had occurred. The control samples, with pH adjustment using NaOH only, exhibited initial brightness changes of "0.5% ISO. After exposure to 7 hours of direct outdoor sunlight, all samples exhibited a significant loss of brightness of 10-14 ISO points as compared to the initial brightness after chemical addition. Following this short term exposure to direct sunlight irradiation, the samples were stored in the dark, and brightness recovery was monitored over the next 19 weeks, to examine if the peroxyborate species loaded on the mechanical pulp could, *in situ*, bleach light-induced chromophores produced by outdoor irradiation, as discussed by Liu and Argyropoulos (14).

The long term brightness recovery was most pronounced for the 3% borax/3% H₂O₂ combination at a pH of 10.8. In fact, the ISO brightness (71.6%) of the handsheet after 19 weeks of dark storage came within 0.7% ISO of the initial handsheet brightness before the experiment was started. The brightness of this handsheet immediately after sunlight exposure was 62.4% ISO, which meant that the 3% borax/ 3% H₂O₂ treatment allowed recovery of over 9% ISO points of brightness during the 19 week dark storage period. Under the same experimental conditions, the handsheets treated with only 3% H₂O₂ recovered 5.5% ISO to reach a value of 68.5% ISO after 19 weeks, while the pH adjusted control regained 4.3% ISO to achieve a final brightness of 66.7% ISO after the 19 weeks of dark storage (**Figure 1**). This data shows the marked improvement that the addition of a small (molar) quantity of borax has on the long term brightness characteristics of the pulp, perhaps due to the ability of the peroxyborate species to perform solid state bleaching over an extended period of time (14).

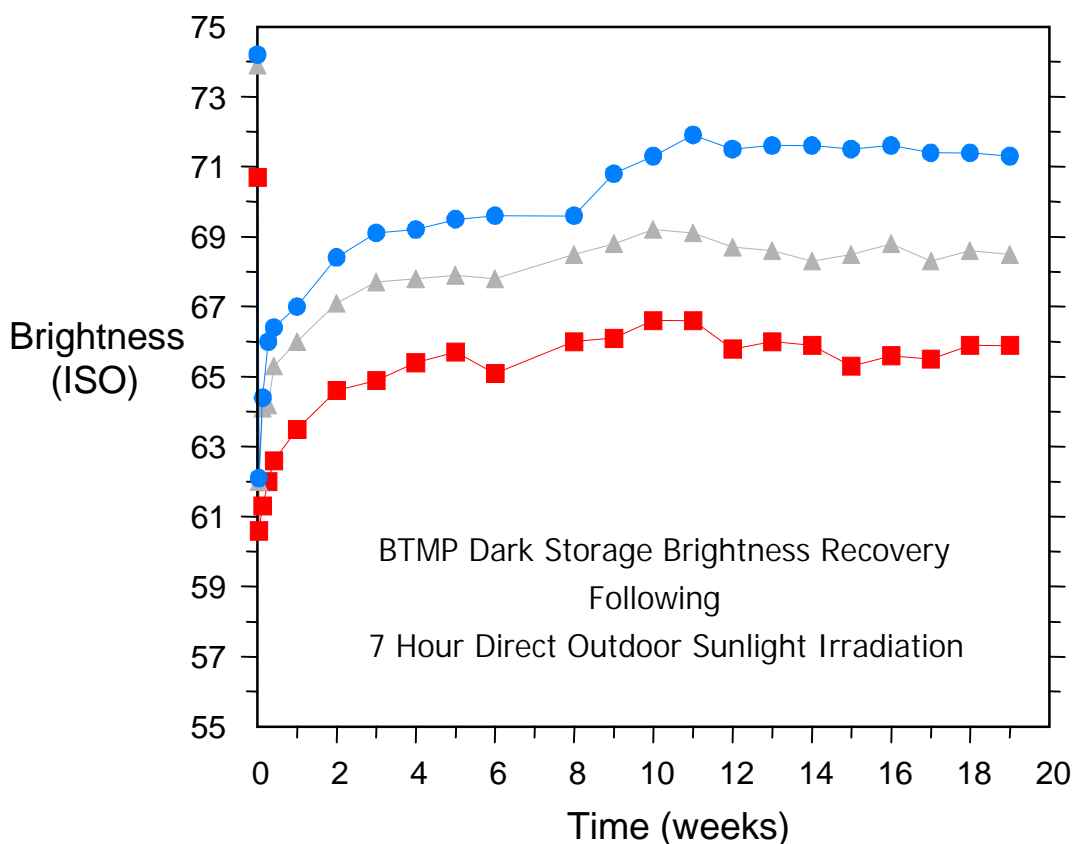


Figure 1. Dark storage ISO brightness recovery of BTMP exposed to 7 hours of direct sunlight with application of 3% H₂O₂ (▲), 3% H₂O₂/3% borax (●), and control (■).

Another laboratory experiment was established to study the effect of exposure of BTMP handsheets with 6 hours direct indoor fluorescent light followed by 18 hours of darkness, during which time solid state bleaching and brightness recovery may occur. The samples were contained in photoreactors for 16 weeks, and were exposed to irradiation of 250 fc, which would approximate the irradiation in a brightly lit room. This experiment was performed so as to simulate the everyday use of paper. The samples were coated with aqueous solutions, at a pH 10.8, of (by weight) 3% H₂O₂, and a combination of 3% H₂O₂/3% borax, with a control sample pH adjusted using NaOH.

Again the addition of 3% H₂O₂ or 3%/3% H₂O₂/borax solutions adjusted to pH of 10.8 increased the initial brightness of the BTMP handsheets 2.5-3.0% ISO, while for the pH control sample, the initial brightness dropped 0.3-0.5% ISO points after the addition of NaOH. The general trend was that as time proceeded the brightness of the samples decreased. There was observed, however, fluctuations in the brightness as the samples were exposed alternately to light and darkness. Following a period of 6 hours of light exposure, the 3% H₂O₂/3% borax treated handsheets regained as much as 1.5% ISO in the subsequent 18 hours of darkness, during the first few days of the experiment (**Figure 2**). Liu and Argyropoulos (14) explain this phenomenon as being caused by the formation of perborate esters which can act as slow solid-state oxidizing bleaching agents capable of partially restoring, in the dark, the brightness losses induced on mechanical pulps during direct outdoor and indoor irradiation. This brightness recovery was also observed for the 3% H₂O₂ treated samples, but to a lesser extent (approximate maximal gains of 1.0% ISO). Interestingly, the pH adjusted control, and a blank control also exhibited some dark exposure brightness reversion (< 0.5% ISO).

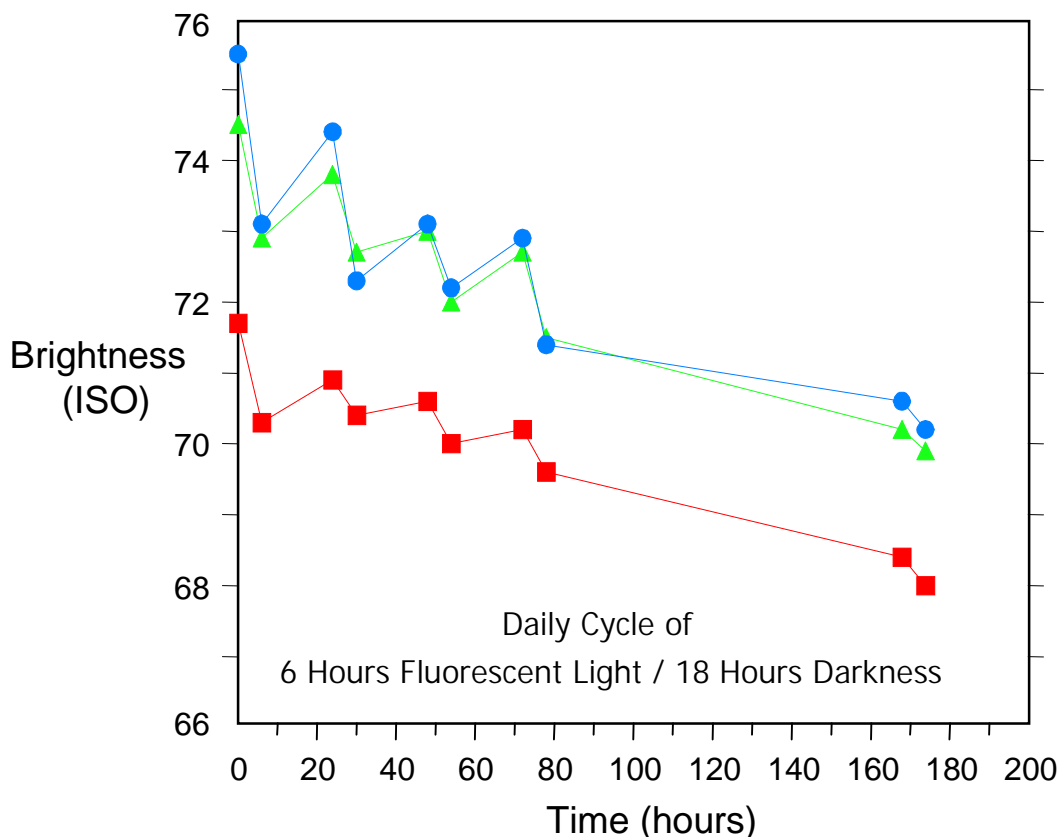


Figure 2. ISO brightness fluctuations of BTMP exposed to 6 hours of fluorescent light followed by 18 hours darkness on a daily basis with application of 3% H₂O₂ (▲), 3% H₂O₂/3% borax (●), and control (■).

By the end of the 16 week experiment, the average brightness of the 3%/3% H₂O₂/borax treated BTMP handsheets was 50.0 "0.1% ISO, and for the 3% H₂O₂ alone treated handsheets the average final brightness was 46.6 "0.2% ISO. The average final brightness of the pH control sample was 45.0 "0.2% ISO, whereas a blank sample with no pH adjustment was 43.0 "0.2% ISO. The brightness stability improvement of the peroxyborate sheets was therefore 16% compared to the control. The results for the trial are summarized in (**Figure 3**), and indicate that the addition of a relatively small amount (on a molar basis) of borax to the hydrogen peroxide greatly increases the long term brightness stability of the BTMP handsheets exposed to simulated ambient light conditions.

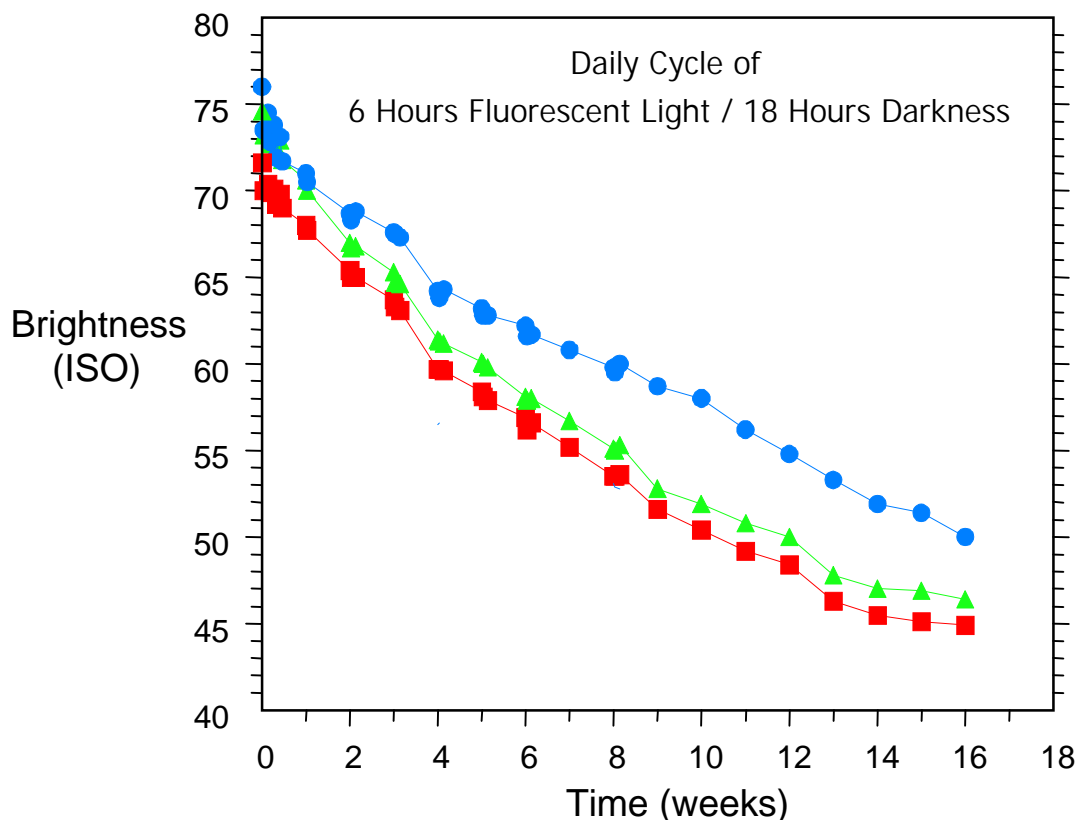


Figure 3. ISO brightness of BTMP exposed to 6 hours of fluorescent light followed by 18 hours darkness on a daily basis with application of 3% H₂O₂ (▲), 3% H₂O₂/3% borax (●), and control (■).

Pilot scale paper making trial

Since the data from the borax/alkaline hydrogen peroxide long-term laboratory irradiation studies was promising, the next logical step in the study of the applicability of this system would be to perform a pilot scale trial using reduced scale paper making equipment. The laboratory results needed to be substantiated in a pilot trial before proceeding with requests for mill trials as this type of pilot scale trial is the only way to determine if this application can be performed at an industrial scale. A pilot trial was undertaken at the Pulp and Paper Research Institute of Canada (PAPRICAN) in Pointe Claire, Québec.

Initially, the desired pickup of the borax and hydrogen peroxide was to be 3% (w/w) for both compounds, as was in the laboratory experiments. Tests undertaken at PAPRICAN indicated that the maximum practical borax application on the upper side of the wet web would be 1.5% w/w, based on the solubility of the borax stock solution, although 3% (w/w) H₂O₂ would be applied during the pilot trial as in the laboratory trials. Four sets of coatings experiments were planned. They were:

- i) 3% H₂O₂ at pH= 10.5-10.8
- ii) 1.5% Borax at pH= 10.5-10.8
- iii) 3% H₂O₂ / 1.5% Borax at pH= 10.5-10.8
- iv) 1.5% H₂O₂ / 1.5% Borax at pH= 10.5-10.8

The pilot trial results confirmed the good brightness retention characteristics exhibited by the laboratory BTMP treated with the (*in situ*) peroxyborate solutions. Unfortunately, an underestimation of the stock application solutions concentrations and/or pumping rate ultimately allowed for a less than desired amount of oxidant to be applied on the wet web. Subsequent analyses for borax (using boron ICP-MS) and for peroxy functionality using the thioanisole test (15), a method developed for the detection of oxidants on solid pulp matrices, indicated that approximately one third of the desired H₂O₂ and borax concentrations actually remained on the paper, and confirmed the calculated concentrations subsequently obtained by flow measurements made by PAPRICAN operators during the coating trials.

The thioanisole test (15) was performed on samples from the same run but dried at the 3 different temperatures. The test confirmed that drying temperature (#160EC) did not affect the initial amount of peroxy species, and potentially the long term bleaching potential. Long term irradiation experiments confirmed that there was no difference in the brightness amongst identical samples whether they were dried at 100EC, 130EC, or 160EC. This data has important implications when considering (*in situ*) peroxyborate as a production scale paper application material, as one concern that may be expressed is whether or not the peroxyborate species would be able to withstand severe paper drying conditions.

Despite the significantly lowered (1.0% H₂O₂/0.5% borax) charges actually applied (compared to the desired 3.0%/1.5% charges), promising long term brightness retention results were realized. The first experiment involved exposing the pilot trial samples to direct outdoor (autumn) sunlight for 7 hours; the samples were then placed in dark storage to allow for brightness recovery (as in the laboratory scale experiments described earlier). After the initial 7 hours of sunlight exposure, brightness values dropped 7-8% ISO, but recovery occurred almost immediately when the sheets were removed from the irradiation source. After approximately 120 days, the brightness recovery (of 2.5% ISO) had reached a plateau for the 1.0% H₂O₂/0.5% borax sample, while all other treated samples, as well as the control, started to again lose brightness over time (**Figure 4**). This suggests that the pathway by which chromophores are removed with the addition of peroxyborate bleaching agents differ from the hydrogen peroxide bleaching pathways, with the peroxyborate species allowing for permanent, irreversible destruction of chromophoric species.

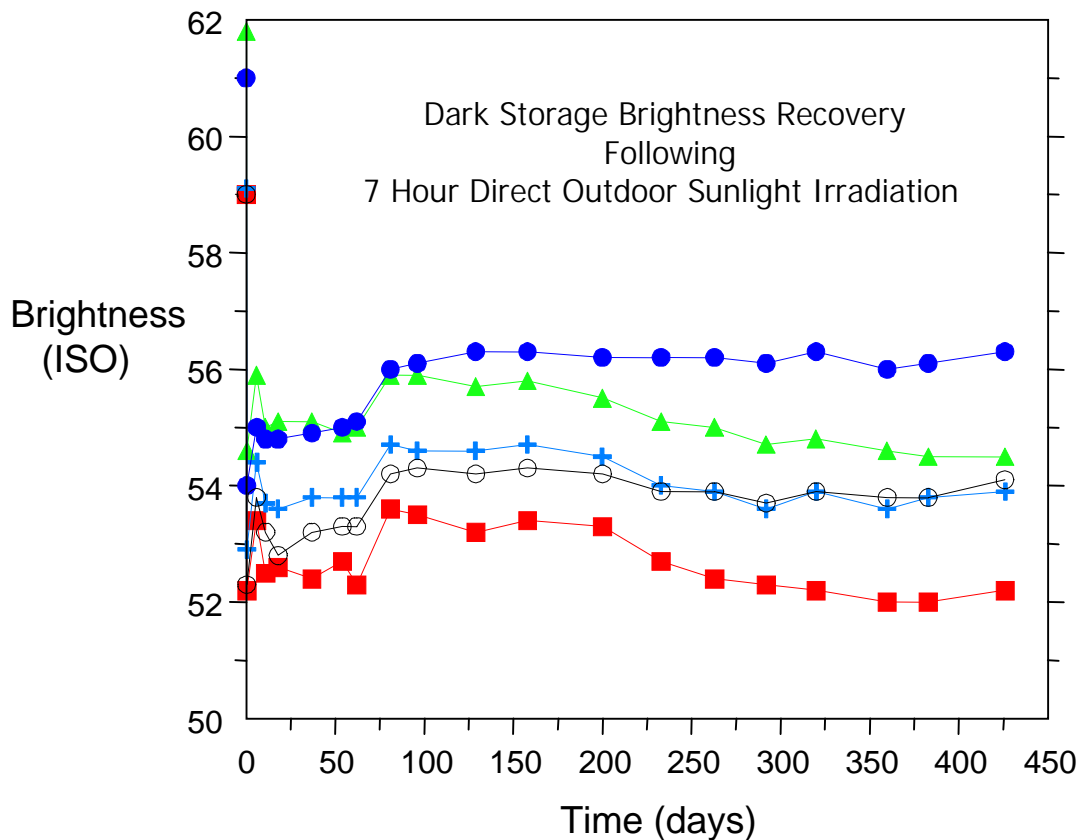


Figure 4. Dark storage ISO brightness recovery of pilot trial paper exposed to 7 hours of direct sunlight with application of 1.0 % H₂O₂ (▲), 0.5% borax (+), 1.0% H₂O₂/0.5% borax (●), 0.5% H₂O₂/0.5% borax (○), and control (■).

In an effort to simulate the effect of typical day-to-day handling of paper products, samples (5 cm x 5 cm) from each run of the pilot trial were placed in an NCE light box under controlled light exposure. The samples were exposed on a daily basis for 6 hours of fluorescent light (250 fc) followed by 18 hours of darkness. At any given time for duplicate trials the brightness values did not usually differ by more than 0.3% ISO. As shown in **Figure 5**, an application of 1.0% H₂O₂/0.5% borax (at a pH of 10.6) provided the best long-term brightness retention after 431 days, with more than 3% ISO higher brightness than non-treated control samples, a stabilization of 10%. In fact, the difference between the control samples= brightness and the *in situ* peroxyborate treated samples= brightness was appearing to get larger as time went on, up to 300 days, at which point the brightness values for the control and treated samples levelled off. Thus it appeared that the rate of brightness loss was slowed for the 1.0% H₂O₂/0.5% borax treated samples as compared to the control. After the 431 day irradiation period, the brightness gains of the 1% H₂O₂ and 0.5% borax treated sheets, compared to the control sheets were 0.5 and 1.3 ISO points respectively, while the brightness gain of the 1% H₂O₂/ 0.5% borax combination was 3.0 ISO points, greater than the sum of the brightness gains of the two additives alone (**Table I**). The addition of borax appears to synergistically improve the bleaching ability of hydrogen peroxide through the formation of peroxyborate species, which in turn allows for a longer bleaching capability.

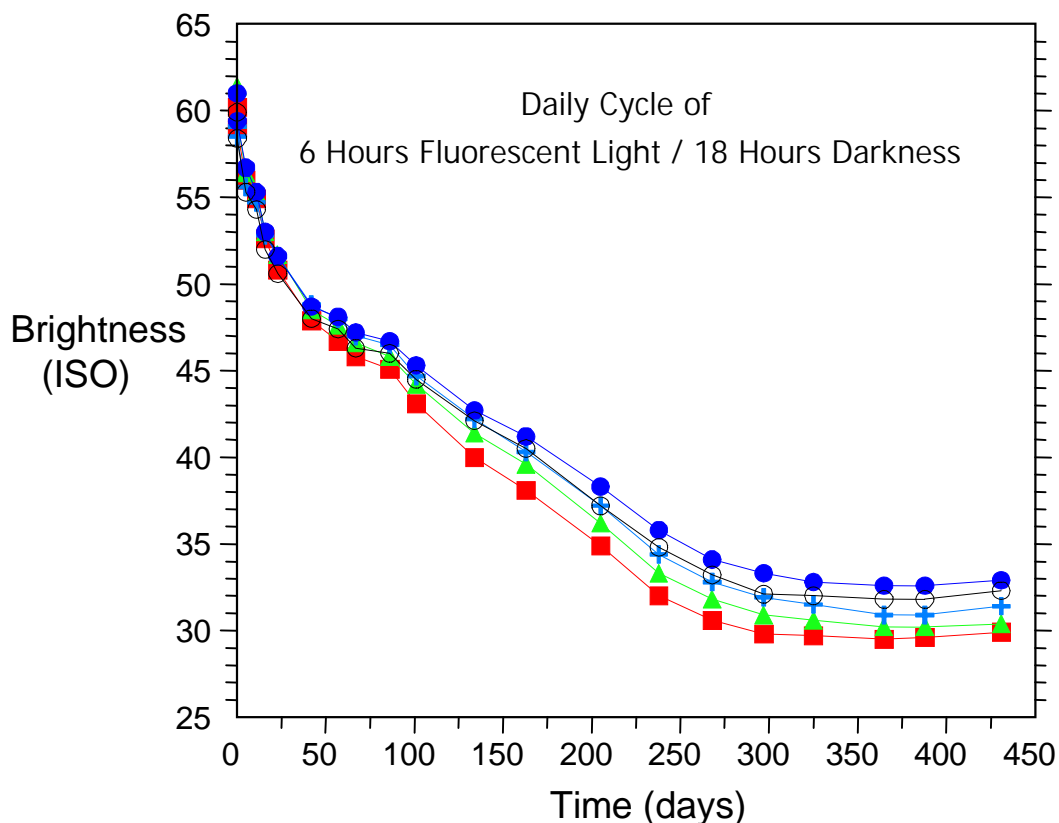


Figure 5. ISO brightness of pilot trial paper exposed to 6 hours of fluorescent light followed by 18 hours darkness on a daily basis with application of 1.0% H₂O₂ (▲), 0.5% borax (⊕), 1.0% H₂O₂/0.5% borax (●), 0.5% H₂O₂/0.5% borax (○), and control (■)

	IMPREGNATION MATERIAL			
	Control	1% H ₂ O ₂	0.5% Borax	1% H ₂ O ₂ / 0.5% Borax
Brightness Gain (% ISO) (compared to control)	0.0 " 0.2	0.5 " 0.1	1.3 " 0.2	3.1 " 0.3

Table I. Brightness gains of various impregnation materials compared to controls after 431 days of exposure to controlled fluorescent light.

Similar trends were observed for the changes in yellowness (b^*) of the coated samples. Paper with the addition of H₂O₂/borax combinations were less yellow (lower b^*) after 431 days compared to paper samples treated with H₂O₂ or borax alone, or with no treatment; these results are statistically significant ($n=3$) as shown in **Figure 6**. These brightness and b^* results indicate that the peroxyborate esters that formed with the carbohydrate components of the pulp allow for a longer retention of bleaching agents, with a resulting transfer of oxidative capability towards the chromophoric groups present in the lignin over a considerably prolonged period of time while exposed to irradiation.

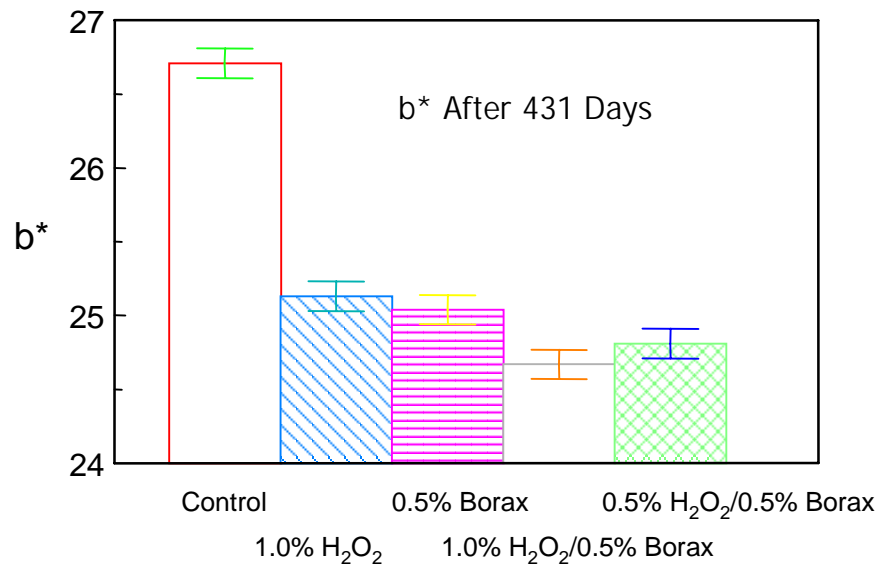


Figure 6. Yellowness (b^*) after 431 days of pilot trial paper exposure to 6 hours of fluorescent light followed by 18 hours darkness on a daily basis.

Ten mechanical property tests were performed on the control and treated paper samples, to determine if there were any advantages to adding a combination of H₂O₂ and borax as a application material on BTMP. Results of the mechanical property tests showed that there were significant (n=5) increases in breaking length, tensile index, and TEA index for the 1% H₂O₂/0.5% borax treated samples, compared to the borax treated, H₂O₂ treated, and control samples (**Figure 7**). Similar trends were also observed in the elastic modulus, Z-span breaking length, Scott bond, stretch, and MIT fold number tests, although these results were not statistically significant.

The structural characteristics of the coated handsheets dried at 130EC were examined by performing various roughness analyses (PPS, Sheffield, and Bendtsen). These methods do not indicate absolute smoothness (or roughness) but demonstrate the degree of smoothness for comparison. Surface smoothness has an important influence on printing quality, friction, gloss and coating adsorption. No significant differences in smoothness, compared to the control paper, arose from applying different coating solutions. This suggests that samples treated with peroxyborate compounds should not affect the surface characteristics and hence, for example, the printability of paper coated with these materials.

From the results from the first pass of static and kinetic friction tests for each of the coated samples and control, the peroxyborate treated samples did not exhibit any higher coefficients of friction compared to the other treated samples and control. As well, specular gloss tests indicated that the surface quality of the BTMP (and thus printing quality) does not appear to be compromised with the addition of the peroxyborate species. These results imply that peroxyborate coated paper sheets will not be detrimentally affected from a print processing point of view.

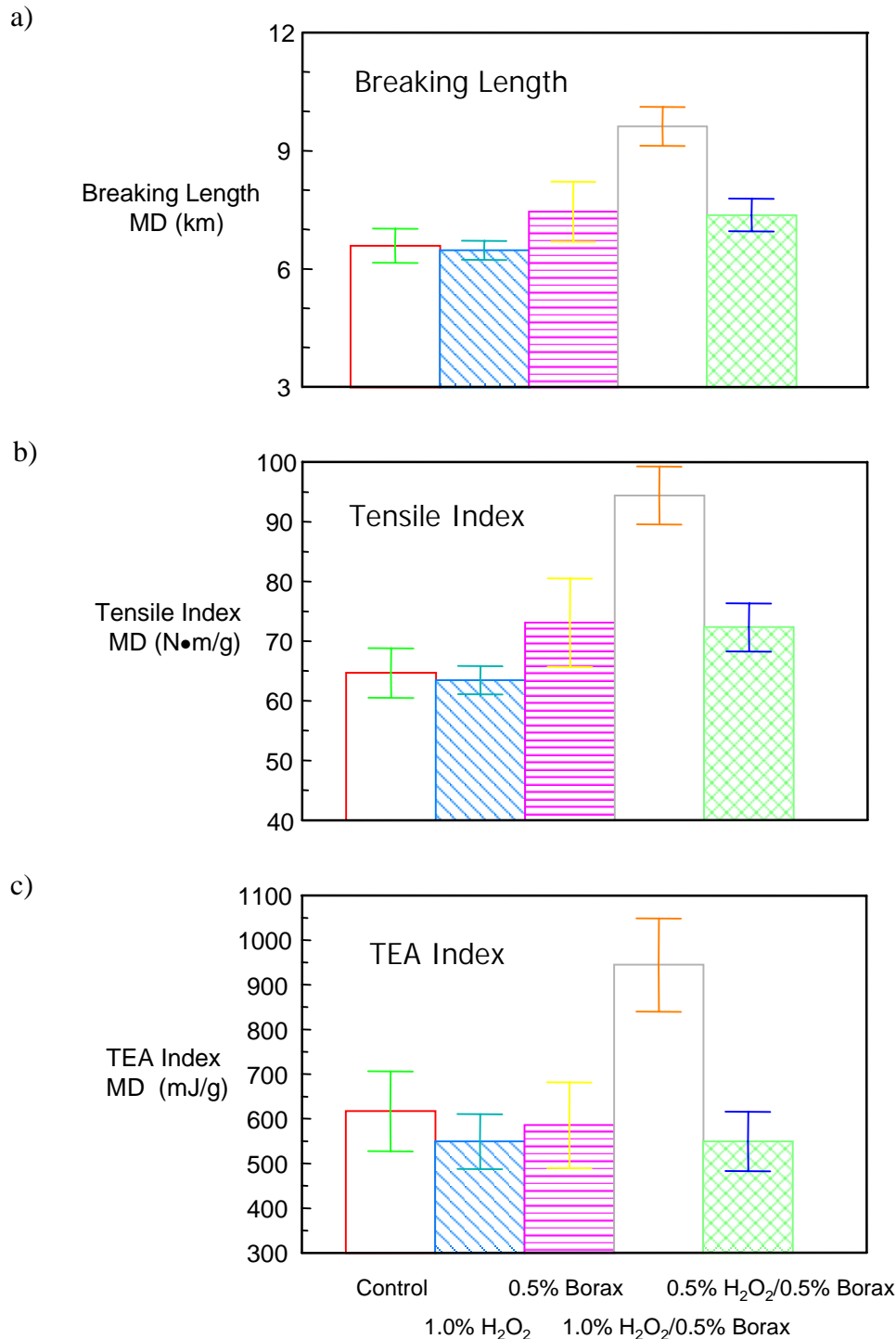


Figure 7. Mechanical property characteristics of the pilot trial paper as shown by breaking length (a), tensile index (b), and TEA index (c).

CONCLUSIONS

This paper has focussed on the use of aqueous active-oxygen bleaching coating agents for thermomechanical pulp. These agents were examined in both laboratory brightness reversion irradiation experiments and in a pilot scale mill trial undertaken at PAPRICAN, Pointe Claire, Québec. It is anticipated that this peroxyborate application on thermomechanical pulp may provide economic advantages to mills experiencing storage reversion problems.

In a series of laboratory irradiation experiments, it was shown that an application of a combination of aqueous alkaline hydrogen peroxide and borax, producing peroxyborates *in situ*, was quite effective at both increasing the initial brightness of bleached softwood TMP sheets as well as maintaining the brightness over a long period of irradiation. Preliminary experiments established the optimal loading conditions to be 3% (w/w) for both the hydrogen peroxide and borax, at a pH of approximately 10.8.

In long term laboratory irradiation experiments, black spruce BTMP samples were treated with H₂O₂ alone, borax alone, and with a combination of alkaline H₂O₂ and borax. The results of these experiments indicated that the addition of a relatively small amount (on a molar basis) of borax to the alkaline hydrogen peroxide coating solution greatly increases the long term brightness stability of TMP sheets.

The applicability of the peroxyborate system was further examined by applying the chemicals in a pilot plant trial on reduced scale paper making equipment. The pilot scale trial results did indeed confirm the excellent brightness retention characteristics exhibited in the laboratory scale BTMP (*in situ*) peroxyborate application experiments. Even with a lowered 1.0% H₂O₂/0.5% borax application, promising long term brightness retention results were realized. Long term irradiation experiments, similar to the laboratory scale peroxyborate application trials described above, were performed. The combination of the hydrogen peroxide and borax together produced much better brightness gains/retention than the sum of the brightness gains produced by either compound alone. This synergism indicates the potential this *in situ* formulation of peroxyborates would have in the commercial bleaching of mechanical pulps. Mechanical and structural properties of the peroxyborate treated samples were also measured, with a number of the tests showing significant improvement compared to control samples or samples treated with H₂O₂ or borax alone. Drying temperatures up to 160°C did not affect the long term reversion ability of the peroxygen species, which has important implications when considering perborate as a production application material.

One problem that was identified during the mill trial was that although the aqueous peroxyborate solution could be applied to the wet web, the amount retained on the paper was less than desired. This problem could be corrected by increasing the pumping rate of the chemical application material, or by adding retention aid to increase the retention of the peroxyborate.

Future research work with the *in situ* peroxyborate bleaching system will include a search for a UV screen or photoyellowing inhibition system that would be compatible when combined with the peroxyborates. It is envisioned that this combination of an *in situ* bleaching and brightness reversion inhibition agent, and a photoyellow inhibitor (UV screen), if possible, would provide a synergistic effect against the photoyellowing of thermomechanical pulp.

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