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Chemical technology/modifications

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Physical properties of kraft pulp oxidized by hydrogen peroxide under mildly acidic conditions

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Abstract: The objective of this study is to gain further insight into the compositional and physical changes of kraft pulp caused by oxidation with hydrogen peroxide under acidic conditions. A softwood kraft pulp has been subjected to varying degrees of oxidation. Changes in the composition, as well as resulting physical properties of laboratory sheets were investigated. Findings indicated that the oxidation resulted in an increased carbonyl group content, a decrease in intrinsic viscosity and a change in hemicellulose composition. The oxidized pulps were found to have a reduced water retention value and a slightly reduced tensile strength (4–11 % at the investigated conditions). However, a substantial increase in wet web tensile strength (6–23 %) and wet tensile strength (134–242 %) of the sheets was obtained. The sheets could easily be repulped and an increased wet strength maintained to a large extent, even after recycling of the sheets.

Keywords: carbonyl groups; hydrogen peroxide; kraft pulp; oxidation; wet strength.

Introduction

In order to reduce the use of fossil-based materials, a significant reduction of the use of plastics will need to take place. One approach to achieve this is the replacement of plastics with cellulose fibre-based materials in applications such as packaging or composites used for manufacturing of different devices. However, cellulose fibre-based materials have a major drawback: when subjected to wet conditions, the fibre web loses over 90 % of its initial strength. This is due to the main bonds between the fibres in the web (in terms of strength) being hydrogen bonds, which are quickly broken in aqueous conditions. Because of this, wet strength agents need to be added to cellulose-based products that need to maintain strength, even under moist conditions.

Wet strength agents are able to enhance the strength of the fibre material under wet conditions through two mechanisms: by protecting existing bonds through prevention of fibre swelling, or through the formation of new, covalent, bonds (Andreasson and Wågberg 2009). Apart from wet strength agents, an operation known as internal sizing may also be utilised to reduce the liquids' penetration into the fibre material through treatment with hydrophobic substances (Lindström 2009). One group of commonly used wet strength agents are wet strength resins. They work by adhering to the pulp and forming a network that hinders the swelling of the fibres and the subsequent separation of fibre-fibre contacts (Espy 1995). However, traditional wet-strength resins have potentially negative environmental impacts during their production, due to the emission of adsorbable organic halides (AOX) (Bates et al. 1999). Additionally, wet strength resins make recycling problematic, as fibre materials with added wet strength resins typically need a relatively harsh alkaline or oxidizing treatment to successfully recycle the material (Yang and Luettgen 2020). Due to these disadvantages alternative ways of creating a fibre material with wet strength are being investigated.

Bio-based wet strength agents can be produced from starches or cellulose through oxidation. By oxidizing the hydroxyl groups on the carbohydrate chain, carbonyl groups can be formed. These carbonyl groups can form a

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hemiacetal crosslink with a neighbouring hydroxyl group. As hemiacetals are not as easily interrupted by water as hydrogen bonds, an increased wet strength is achieved. These oxidized carbohydrates are typically obtained using either sodium metaperiodate (Kim et al. 2000) or TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl radical) (Saito & Iso-gai 2005, Jaschinski et al. 1999), the former yields a C2–C3 dialdehyde and TEMPO may yield C6 aldehydes. However, these oxidants have some disadvantages. Sodium metaperiodate is environmentally harmful and TEMPO has economical drawbacks (Serra et al. 2017), making it unsuitable for use in large-scale production.

As a potential alternative to the above-mentioned oxidants, previous studies have successfully employed hydrogen peroxide under acidic conditions as a means of increasing the number of carbonyl groups (Martinsson et al. 2020, Martinsson et al. 2021). These studies have focused on the ability of hydrogen peroxide to introduce carbonyl groups, the effects on fibre composition, degradation of carbohydrates and total charge of the pulp. But the effect on physical properties of a fibre web, such as tensile strength, wet web tensile strength and the drain-ability of a fibre suspension has yet to be investigated. Previous work by Erlandsson et al. (2018) has shown that periodate oxidized cellulose nanofibrils will form hemiacetal crosslinks already in the wet state, if the oxidized carbohydrate surfaces are in close contact, such as during pressing. This suggests that hemiacetals will form as early as during the pressing of the wet fibre web.

Furthermore, it is well known that the repeated recycling of kraft pulp sheets results in a reduction in tensile strength, with a more dramatic loss in strength properties taking place during the initial rounds of recycling (Van Wyk and Gerischer 1982, Nazhad and Paszner 1994). This is typically explained by hornification and reduced inter-fibre bonding. No previous studies have been found that conclude whether the introduction of oxidized groups affect this behaviour. Consequently, the effect of oxidation on the recycling of fibres, or the ability to maintain wet strength after recycling, remains to be investigated.

This study therefore investigates the effect of acidic hydrogen peroxide treatment on key physical properties of the oxidized pulp, such as tensile strength, wet tensile strength and wet web tensile strength. The repulping of laboratory sheets was also carried out in order to demonstrate the fibre treatment during recycling and investigate if the wet strength can be maintained, which is of interest for the recycling of materials produced from oxidized pulps, or for re-pulping of broke.

Materials and methods

Materials

A softwood kraft pulp provided by Södra Cell Värö was used as starting material for the oxidations. Hydrogen peroxide (30 %), sodium acetate (≥ 99 %) and glacial acetic acid were all purchased from Merck and used as received.

Pulp oxidation

15 g of softwood kraft pulp was oxidized at a solid to liquid ratio of 1:40 in an acetate buffer at pH 4, using a varying charge of hydrogen peroxide (50 % or 200 % on pulp). The temperature was kept constant at 85 °C and the residence time used was 90 minutes. The reaction was carried out in a 500 ml jacketed glass reactor (100 mm in diameter) equipped with baffles. The reactor was stirred using a pitched blade impeller with a diameter of 50 mm at 1000 rpm. A circulating heating bath with external temperature control was used to maintain a constant temperature. Once the 90 minutes had passed the reaction was stopped by adding the suspension to a beaker containing 500 ml of cold, deionised water. The suspension was filtered and the filtrate recirculated once, followed by washing with 1000 ml of deionised water. The pulp was then added to 500 ml of deionised water and left for 10 minutes, after which the suspension was filtered, and the filtrate recirculated once. Finally, the pulp was washed with an additional portion of 1000 ml of deionised water pH was adjusted to pH 3.5 using sulphuric acid.

Intrinsic viscosity

The intrinsic viscosity of pulp dissolved in bis(ethylenediamine)copper(II) hydroxide (CED) solution was measured according to SCAN-C 15:62. Prior to dissolution in CED the pulp was reduced using 3 % sodium borohydride at 4 % consistency in order to minimise the effect of degradation at alkaline conditions due to the carbonyl groups. A sample of air-dried pulp was torn into pieces and dispersed in 25 ml of deionised water, after which 25 ml of 1 M CED was added. The sample was conditioned in a water bath set to 25 °C. The intrinsic viscosity was then measured using a capillary viscometer.

Carbonyl content

The total number of carbonyl groups was measured using a method based on the one by Zhao and Heindel (1991).

A never-dried pulp sample corresponding to 0.5 g of oven-dried pulp was added to 100 ml of deionised water, pH adjusted to pH 4 and dispersed thoroughly. After 10 minutes, the suspension was filtered and the sample washed with 100 ml of deionised water, pH adjusted to pH 4. The pulp was transferred to a beaker containing 25 ml of 0.25 M hydroxylamine hydrochloride, pH adjusted to pH 4. The beaker was then placed on a shaking table for 2 hours, after which the suspension was filtered and the filtrate titrated back to pH 4 using 0.01 M NaOH. The pulp was washed with deionised water and placed in an oven at 105 °C for 24 hours for determination of the sample mass. The total number of carbonyl groups were calculated from the amount of titrant used to titrate the filtrate to pH 4 and the oven-dry weight of the pulp.

Total charge

The total charge was measured according to SCAN-CM 65:02. A pulp sample corresponding to 1 g oven-dry weight was protonated using 0.1 M HCl at 1% concentration for 15 minutes. After protonation the sample was washed repeatedly with deionised water until the filtrate measured a conductivity less than 5 $\mu\text{S}/\text{cm}$. The sample was then transferred to a beaker containing 490 ml of deionised water and 10 ml of 0.05 M NaCl. The suspension was titrated using 0.05 M NaOH using addition steps of 0.1 ml, 30 seconds between each addition. The conductivity was plotted against the added sodium hydroxide, and the total charge was calculated using the amount of sodium hydroxide added at the second intersection point. After titration, the suspension was filtered onto a pre-weighed filter and the sample was placed in an oven at 105 °C for 24 hours for determination of sample weight.

Compositional analysis of pulp

The composition of the pulp was analysed in terms of Klason lignin, acid soluble lignin (ASL) and monomeric sugar content after acid hydrolysis. An oven-dry sample of 200 mg was subjected to complete acid hydrolysis with sulphuric acid using a method based on the work of Theander and Westerlund (1986). After hydrolysis the sample was filtered and the insoluble fraction was considered to be Klason lignin. The hydrolysate was then used for the determination of ASL with an Analytik Jena Specord 150 UV spectrometer at a wavelength of 205 nm, using an absorptivity constant of 110 $\text{dm}^3/\text{g cm}$. The hydrolysate was further analysed for monomeric sugar content using a Dionex ICS-5000 equipped with CarboPac PA1 columns

using NaOH/NaAC (Aq) and NaOH (aq) as eluents, and an electrochemical detector for detection. Chromeleon 7, version 7.1.3.24.25 was used as software.

Sheet forming

As never-dried pulp was used, sheets with a grammage of 370 g/m^2 were first prepared and subjected to a drying procedure that simulates the drying process (airborne web dryer) of a pulp mill producing market pulp. Sheets were prepared using a sheet former, after which the wet sheets were stacked with 3 blotters and an ebonite plate in between each new sheet and pressed for 10 minutes at 50 bar. The blotters were then replaced and the pressing repeated. After the second pressing the blotters were removed and the sheets placed in a drying cupboard set at 90 °C and 50 % relative humidity for 60 minutes. These sheets were then used to produce the stock for the measurement of water retention value, Schopper-Riegler, as well as for production of laboratory sheets, as seen in Figure 1.

Laboratory sheets were prepared according to ISO 5269-1. No beating of the pulps was performed. The previously prepared pulp sheets were defibrillated in a standard pulp disintegrator for 30 000 revolutions. The pulp suspension was transferred to a stirred tank and diluted to a concentration corresponding to a sheet grammage of 60 g/m^2 and sheets were formed using a sheet former (Finnish type, Lorentzen & Wettre, Sweden). The wet sheets were stacked between blotters and ebonite plates and pressed at 4 bar for 5 minutes, after which the blotters were changed and another pressing at 4 bar for 2 minutes was conducted. The pressed sheets were transferred to a conditioned room and air dried on the ebonite plates for 24 hours.

Tensile strength

The tensile strength was tested according to ISO 1924-3:2011. 15 mm wide test strips were cut from laboratory sheets with a target grammage of 60 g/m^2 . The test strips were tested using a Lorentzen & Wettre horizontal tensile tester, with a separation speed of 100 mm/min.

Wet tensile strength

Wet tensile strength was tested in accordance with ISO 12625-5:2017, using a Lorentzen & Wettre horizontal tensile tester. The test strips were cut to 50 mm width and subjected to a soaking time of 15 seconds, followed by a pull of 50 mm/min.

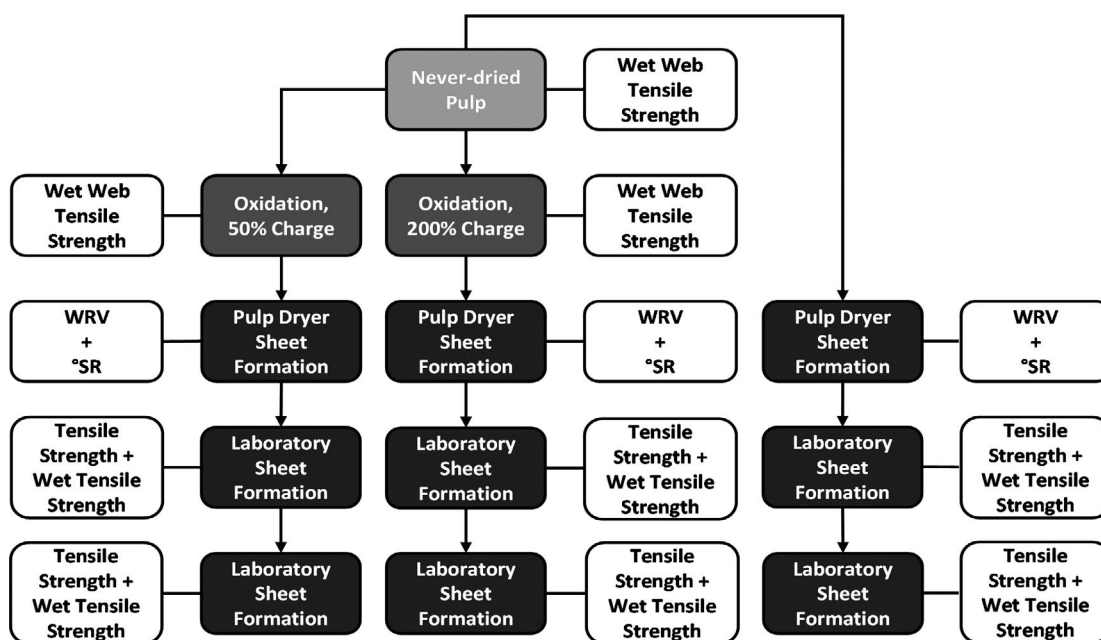


Figure 1: Schematic description of the sheet forming procedure.

Wet web tensile strength

A stock corresponding to a dry sheet grammage of 60 g/m² was prepared and a sheet formed in a sheet former, according to ISO 5269-1:2005. After transferring the sheet from the sheet former, the moist sheet was placed in a stack consisting of a press felt, a plastic sheet, 2 blotters, the moist sheet (on a blotter), 3 blotters, a plastic sheet and a press felt. The stack was placed in a press set to 4 bar and pressed for a set time. The press time was varied in order to vary the dry content of the sheet. Immediately after pressing, the sheet was removed from the stack, placed between two plastic sheets and moved to a conditioned testing room. While still between the plastic sheets, the sheet was cut into 3 strips, 50 mm wide and the tensile strength was tested immediately after on a Lorentzen & Wettre tensile tester. A total of 9 test strips were tested for each pressing time. Press times were 3 minutes, 5 minutes and 8 minutes. All pieces of the sheet were collected immediately after testing and the moist weight was noted. After 24 hours at 105 °C the dry weight was noted and the dry content at the time of testing was calculated.

Water retention value

Water retention value (WRV) was measured according to ISO 23714:2015, however a centrifugation time of 15 minutes, instead of 30 minutes was used. A test pad was formed from a stock with a concentration of 2 g/L by dewatering through a glass-fibre filter. The test pad was trans-

ferred to a test-pad-holding unit and centrifuged for 15 minutes at 3000 g. The moist weight after centrifugation was noted and the sample was placed in an oven at 105 °C for 24 hours. The WRV was calculated using the moist weight after centrifugation and the oven-dry weight.

Schopper-Riegler method

The drain-ability of the pulp was evaluated through the Schopper-Riegler (SR) method, according to ISO 5267-1:2000. 1000 ml of a stock of 2 g/L was poured into the drainage chamber of a Schopper-Riegler apparatus and the SR number was found based on the amount of drained water.

Fibre image analysis

A sample of never-dried pulp was dispersed in deionised water at a concentration of approximately 50 mg/L. The suspension was analysed using a Kajaani FS300 Fibre Analyzer.

Results and discussion

Pulp analysis

The reference pulp and the oxidized pulps were analysed for carbonyl content, total charge and intrinsic viscosity,

Table 1: Results from analysis of the reference pulp and the oxidized pulps.

	Residence Time (min)	Temperature (°C)	pH	H ₂ O ₂ Charge on Pulp (%)	Total Number of Carbonyl Groups (μmol/g)	Viscosity (cm ³ /g)	Total Charge (μmol/g)
Reference	–	–	–	–	8.875	800.45	35.86
Low Oxidation	90	85	4	50	28.11	559.93	32.82
High Oxidation	90	85	4	200	66.40	307.85	36.33

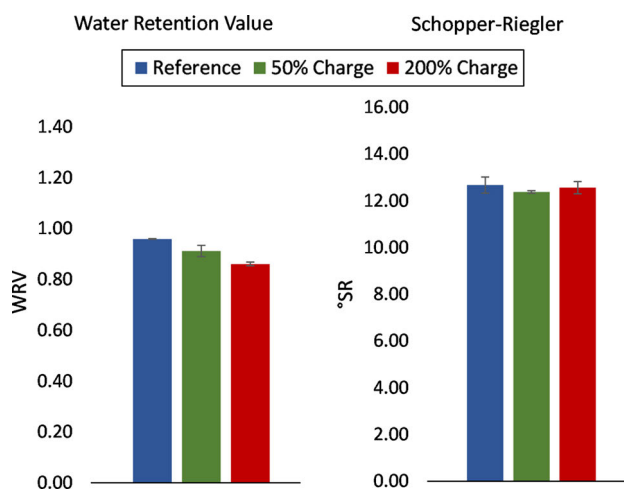
as seen in Table 1. The oxidation resulted in an increased number of carbonyl groups as well as a severe reduction in intrinsic viscosity. The total charge was, however, found to be not markedly affected. This implies that the oxidation mainly results in the formation of aldehydes and/or ketones, rather than further oxidation of formed aldehydes into carboxyl groups. Alternatively, the carboxyl groups may form on shorter carbohydrate fragments that are solubilised, and therefore do not remain in the pulp fraction.

Furthermore, the never-dried pulps were analysed using a fibre image analyser, resulting in the fibre dimensions and fines content found in Table 1. The oxidation does not appear to result in any apparent changes to the fibre dimensions or fines content.

The compositional analysis of the pulps revealed a rather modest decrease in hemicellulose content with increasing oxidation, seen in Table 3. The reduction in hemicellulose content can likely be explained by solubilisation and/or the partial oxidation of the hemicellulose fraction, resulting in fragments that are not monomeric, non-oxidized sugars after the hydrolysis and, thus, not detected with the analytic method used in this study or separated from the pulp during the washing. The rather modest decrease of hemicelluloses may be explained with that the hemicelluloses may be adsorbed, and thus less accessible, on internal/external fibre wall surfaces where cellulose has been exposed during the delignification process (Köhnke 2010).

Physical testing

The pulps were tested for water retention value (WRV) and Shopper Riegler (°SR), as seen in Figure 2. The oxidation was found to result in a reduction of WRV. An explanation for this behaviour may be the formation of intra-fibre crosslinking through the action of the introduced carbonyls. This potentially limits the swelling and water uptake and results in a reduction in WRV. Similarly, an increased hornification, possibly due to the removal of hemicelluloses (see Table 3), may also result in a reduction in

**Figure 2:** Water retention value (left graph) and Shopper-Riegler (right graph), for the reference, and the two oxidized pulps.**Table 2:** Data from fibre image analysis of the reference pulp and oxidized pulps (all never-dried). Length-weighted fibre length, width and fines percentage is presented.

	Reference	50 % H ₂ O ₂ Charge	200 % H ₂ O ₂ Charge
Fibre length (l) (mm)	2.41	2.35	2.39
Fibre Width (l) (μm)	30.3	30.4	30.2
Fines (l) (%)	4.66	4.85	4.73

WRV (Oksanen et al. 1997). However, the drain-ability/de-watering rate was found to be unaffected since the result of the Shopper-Riegler measurements of the different pulps were within the experimental error. This is in line with the results presented in Table 2, which demonstrate that the fibre dimensions and content of fines was virtually unchanged.

Wet web tensile strength

The wet web tensile strength was tested at varying pressing times in order to investigate the effect of oxidation of

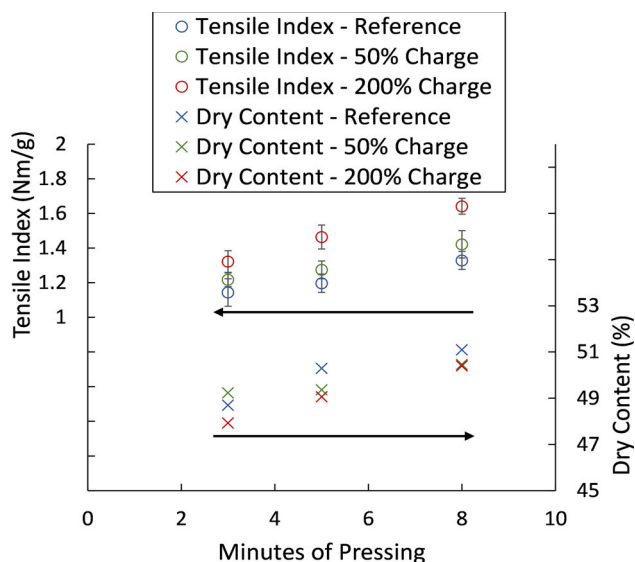


Figure 3: Wet web tensile index of the reference pulp and the two oxidized pulps as a function of the pressing time, as well as the resulting dry content.

Table 3: Composition of the reference and oxidized pulps based on monomeric sugars, Klason lignin and acid soluble lignin (ASL).

	Reference	50 % Charge	200 % Charge
Arabinose (%)	0.46	0.45	0.42
Galactose (%)	0.16	0.15	0.13
Glucose (%)	82.4	81.6	82.7
Xylose (%)	6.44	6.24	5.96
Mannose (%)	5.51	4.90	4.95
Klason (%)	0.70	0.70	1.00
ASL (%)	0.54	0.52	0.57
Total (%)	96.2	94.5	95.7
Undetected (%)	3.82	5.50	4.30

the pulp on paper machine runnability, see Figure 3. Interestingly, the oxidized pulps have a lower resulting dry content, compared to the reference pulp, except for the shortest pressing time where the 50 % charge has the highest dry content. However, the differences in dry content are relatively small. Despite having a lower dry content, the oxidized pulps exhibit a higher tensile index. During the formation of the fibre network, at dry contents below 50 %, the strength of the fibre network is mainly dependent on inter-fibre capillary forces and entanglement friction (van de Ven 2008, Lindqvist 2013). With an increasing dry content, a resulting increase in wet web tensile strength is observed. As the wet web tensile strength is higher for the oxidized pulps compared to the reference, at lower dry contents, the increase is likely the result of an additional phenomena. The introduced carbonyl groups are likely form-

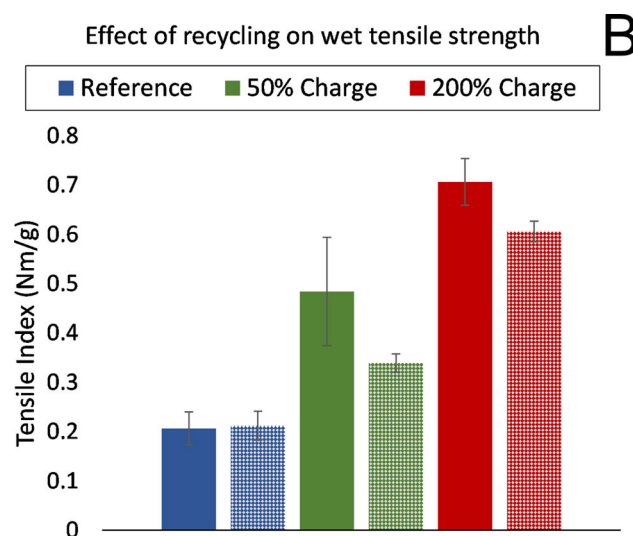
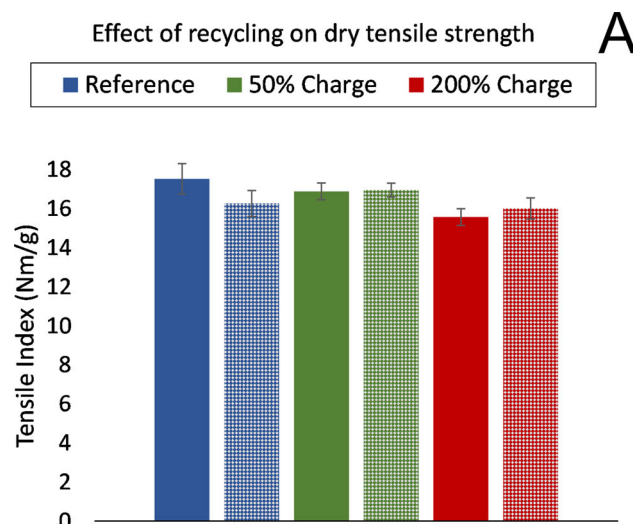


Figure 4: (A) Tensile index for the reference pulp and the two oxidized pulps. The patterned bars represent the once-recycled sheets. (B) Wet tensile index of the reference pulp and the two oxidized pulps. The Patterned bars represent the once-recycled sheets.

ing hemiacetal crosslinks, which have been found to form early on, in the wet state (Erlandsson et al. 2018). The obtained enhancement of wet web strength could potentially enhance the runnability of the oxidized pulp in a paper machine, which could also be accomplished by partly mixing in oxidized pulps in pulps with poor runnability.

Tensile strength of laboratory sheets

In Figure 4, results from strength test on laboratory sheets and once-recycled laboratory sheets are presented. The tests on the once-recycled sheets were conducted in order

to evaluate how the wet tensile strength was affected by the recycling of the fibres through repeated defibrillation, sheet forming, pressing and drying. In regard to the recycling of the sheets, the laboratory sheets could be defibrillated in water and, ocularly, no issues with fibre aggregation could be seen. This easy recycling is potentially beneficial for both recycling of finished products and the recycling of broke.

The oxidation resulted in a minor reduction in dry tensile strength: about a 4 % loss in the case of a 50 % hydrogen peroxide charge, and 11 % for the 200 % charge. The reduction may be the result of several phenomena. As there was no difference between reference and oxidized samples observed from the fibre image analysis, the change in dry tensile strength is likely caused by chemical changes as a result of the oxidation. For example, an increased hornification for the oxidized samples which could also cause the previously mentioned reduction in WRV for the oxidized pulps. This will, in turn, result in a reduction in relative bonded area (RBA), and thereby a reduced tensile strength (Nazhad 2005, Hubbe et al. 2007). The reduced strength of the individual fibres as a result of the degradation of carbohydrates may also partially contribute to the reduced tensile strength. When recycling the reference laboratory sheets, a reduced tensile strength was observed. However, regarding the recycled oxidized sheets, tensile strength seems to be unchanged, or even slightly improved. The standard deviation for these measurements is, however, relatively large, compared to the absolute values, making it difficult to draw quantitative conclusions from the data. Increase in tensile strength due to recycling has previously been reported for unbeaten chemical pulps, where it is attributed to a reduced curl after repeated recycling (Howard and Richard 1992).

The wet tensile strength was significantly enhanced by the oxidation, as seen in Figure 4, which is likely a direct result of the formation of inter-fibre hemiacetals originating from the introduced carbonyl groups (Saito and Iso-gai 2005), as discussed above. The laboratory sheets produced from the recycled sheets had an improved wet tensile strength, compared to the reference pulp. However, compared to the sheets prior to recycling, a decrease in wet tensile strength was observed. Similar to the results from dry tensile tests of the reference pulp, an increased hornification due to the recycling will likely result in a reduced RBA. In turn, this may cause a reduced amount of inter-fibre hemiacetal formation and thereby a reduced wet tensile strength after recycling.

Conclusion

The oxidation with hydrogen peroxide under weakly acid conditions was found to successfully enhance the wet web strength of the material, as compared to the unoxidized reference pulp. At identical pressing times, the resulting dry content of the oxidized material was slightly lower than that of the reference material, yet a higher wet web tensile strength was achieved. The enhancement of wet web tensile strength indicates formation of inter-fibre hemiacetals.

An enhanced wet tensile strength was also observed for the oxidized material. Recycling of laboratory sheets demonstrated that the sheets could easily be disintegrated and reformed into new sheets, and that the enhanced wet tensile strength could be maintained to some degree.

The oxidation resulted in a decrease in tensile strength of between 4–11 % for the two oxidized pulps, compared to the reference pulp. When recycled, a decrease in dry tensile strength was observed for the reference pulp. However, the same trend was not noted in regard to the oxidized pulps.

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Conflict of interest: The authors declare that they have no conflict of interest.

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