Effect of Chemical Crosslinking on the Biodegradation Rate of Kraft Paper

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Wood-based paper fibers are inherently biodegradable. In contact with moist soil and in compost, papermaking fibers are readily broken down by soil microbes. Resistance to biodegradation is needed, as paper is used for special applications such as mulching in agriculture and forestry, the coating of construction materials, and for packing and wrapping under conditions where packaging materials may be exposed to contact with moist soil or other type microbial active contamination. A preceding study showed that paper chemical crosslinking with glyoxal, citric acid (CA), or methylated 1,3-dimethylol-4,5-dihydroxyethylene urea (mDMDHEU) results in substantially improved paper wet strength and lower paper water absorbency. The present study examined the efficiency of chemical crosslinking treatments with CA and with mDMDHEU to decrease the biodegradation rate of laboratory paper sheets and a sack paper, both made of kraft fibers. The biodegradation was examined using a 48-h enzymatic degradation test and a 2-month soil burial test. The results indicate that chemical crosslinking is an effective non-biocidal method for making sulphate kraft paper more resistant to biodegradation. In some end-uses, improved resistance to biodegradation, along with improved paper wet performance, can enhance paper performance comparable to plastic films.

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INTRODUCTION

Like all plant fibers, wood-based papermaking fibers are inherently biodegradable. In moist soil and in compost, biodegradation of paper fibers takes place *via* action cellulolytic and lignolytic enzymes produced by soil microbes (Fedorak 2005; Zambrano *et al*. 2020). From an environmental effect point of view, paper biodegradability is usually considered an advantageous characteristic, as it makes paper waste and litter less harmful for the environment compared to plastics, which the soil microbes are not capable of attacking and biodegrading. Paper biodegradability also enables composting of paper trash bags and kitchen paper along with organic waste. There are some special paper applications, such as mulching papers, building papers, insulation papers, and exterior packaging and wrapping papers, where a certain amount of resistance to biodegradation is desired (Crandall 1954; Vind 1967; Pasanen *et al*. 2000; Wasserbauer 2004; Jerusik 2010; Ahokas *et al*. 2014). In those cases, novel non-biocide solutions for improving paper resistance to biodegradation could improve paper competitiveness compared to plastic films.

The tendency of paper fibers to biodegrade in moist soil depends on the fibers' accessibility to water and on the degrading enzymes produced by the soil microbes (Fedorak 2005; Zambrano *et al*. 2020). In moist paper water molecules swell and open the fiber cell walls and carry the decomposing enzymes to contact with cellulose and lignin (Zambrano *et al*. 2020). Usually, papers made from chemical pulp fibers such as kraft (or "sulphate") pulp fibers biodegrade faster in soil than those made from mechanical fibers such as thermomechanical pulp (TMP) or groundwood pulp (GW) fibers. This is supposed to be due to the more porous structure of the delignified chemical pulp fibers, thus providing access to both water and enzymes to penetrate fiber walls (Andrady *et al*. 1992; Vikman *et al*. 2002; Kwon *et al*. 2021). In addition, soil microbes preferentially attack cellulose over lignin. Biodegradation of cellulose yields energy-rich monosaccharides including glucose and xylose, which the microbes can use for food and growth. The rate of the paper biodegradation process is also affected by ambient conditions such as moisture and temperature, and availability of oxygen and nitrogen (Azim *et al*. 2018; Margida *et al*. 2020).

Paper can be protected against biodegradation using biocides or by coating the fibers or the paper with non-biodegradable, impermeable materials, such as polyethylene (PE) plastic film. An advantage of biocides is that they can also impede the biodegradation of external contaminants such as pollen, dust, and grease, attached on paper surfaces, and in that way suppress visible fungus growth on paper products. The downside of the use of biocides in paper manufacturing and converting is the risk of harmful effects of the biocides, which are inherently toxic to paper mill workers, consumers, and the environment (Grandal 1954; Murtoniemi *et al*. 2003; Jerusik 2010). The feasibility of the coating depends above all on the end-use, manufacturing costs, and the paper properties sought. For example, mulching papers, which can be used as a biodegradable option to non-biodegradable plastic mulching films, should resist biodegradation and maintain their physical integrity throughout the cultivation season but also biodegrade fully afterwards, as plowed into the soil, to such an extent that no harmful cumulative paper littering of soil takes place. Therefore, paper mulches should preferably not contain any nonbiodegradable plastic film coatings or other nonbiodegradable manmade constituents (Ahokas *et al*. 2014; Haapala *et al*. 2014).

One way to increase the biodegradation resistance of cellulosic fibers is *via* the chemical derivatization of cellulose (Reese 1957; Glasser *et al.* 1994; Leppänen *et al.* 2020; Erdal and Hakkarainen 2022). In chemical derivatization, cellulosic hydroxyl groups are substituted by other chemical groups such as acetyl or methyl groups. Because of the substitution, the cellulolytic enzymes, which are highly substrate specific, are less capable of catalyzing the hydrolysis of the cellulose backbone and thus degrade the cellulose derivative. In accordance with early conclusions by Reese (1957), the study by Leppänen *et al*. (2020) showed that most cellulose derivatives, whose degree of substitution (DS) per cellulose glycosidic unit exceed approximately a value of 1.0, show "poor biodegradability". This high DS means, for example, for cellulose acetylation, over 20% calculated weight gain (WG%) of the substrate. Chemical derivatization has been traditionally applied to the manufacture of such specialty papers, for which unique technical properties of the paper such as decreased water absorbency, increased electrical resistance, *etc*., are utilized (Ward 1973).

The present study examined the effectiveness of paper chemical crosslinking treatment on the biodegradability of laboratory handsheet paper (HP) made from Nordic bleached softwood kraft pulp (NBSK) and on a commercial sack paper (SP) made from

Nordic unbleached softwood kraft pulp (NSK). In the treatment, covalent intra- and inter-fiber chemical bonds ("crosslinks") are formed between adjacent cellulose molecules. Unlike cellulose derivatives, which are linear polymers, chemical crosslinking results in three-dimensional cellulosic structures with quite different properties from un-crosslinked cellulosic polymers (Ward 1973).

Chemical crosslinking is used customarily for cotton fabric finishing to retain fabric smoothness and dimensions as the fabric is washed, dried, and worn (Schindler and Hauser 2004; Dehabadi *et al*. 2013; Choudhury 2017). In addition, a study by Smith *et al*. (2021) showed that crosslinked cotton fabric had better resistance to biodegradation compared to non-crosslinked reference fabric.

A recent study by Korpela *et al*. (2023) showed that chemical crosslinking of paper following an ordinary cotton fabric crosslinking method, called the pad-dry-cure method, results in substantially improved paper wet strength and decreased water absorbency. Differing from the non-crosslinked cellulose derivatives, the formed inter-fiber crosslinks can improve the paper wet strength and thus maintain the paper strength and physical integrity as the paper is in contact with moist soil or is otherwise wetted. The crosslinking agents used were citric acid (CA) and methylated 1,3-dimethylol-4,5-dihydroxyethylene urea (mDMDHEU) with appropriate catalysts. The CA is a natural and relatively low-cost crosslinking agent for cellulose, and mDMDHEU is used extensively in industry for cotton fabric crosslinking treatment. The previous study by Korpela *et al*. (2023) showed that compared to CA or glyoxal, crosslinking with mDMDHEU has a less negative impact on paper flexibility.

In the present study, the effect of crosslinking treatments on the paper biodegradability was examined using a 48-h enzymatic degradation test and a 2-month soil burial test. In addition, fungal resistance tests were performed to ensure that the observed effects on paper biodegradability rates were not caused by some unknown biocidal side effects of the used chemical crosslinking agents.

The effects of the treatments on the paper mechanical properties and water absorption were also measured. The chemical crosslinking of cellulose with CA and DMDHEU is shown in Fig. 1.

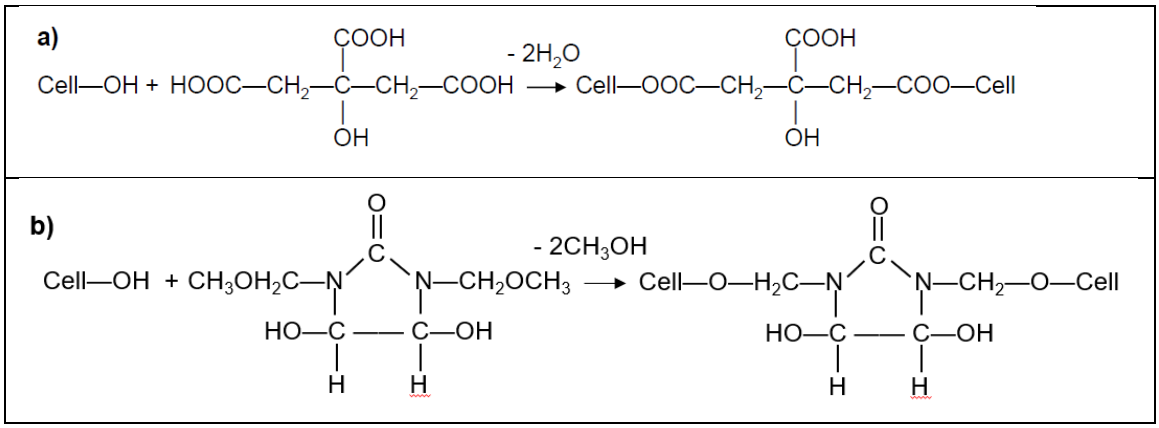

Fig. 1. Crosslinking of cellulose with a) CA (Caulfield 1994) and b) mDMDHEU (Schindler and Hauser 2004)

EXPERIMENTAL

Materials and Preparation of Laboratory Sheets

The NBSK pulp sheets for making laboratory handsheet paper (HP) were obtained from a Finnish pulp mill. The handsheets were made using uncirculated ion-exchanged water, following ISO 5269-1 (2005). The targeted handsheet grammage was 100 g/m^2 at 50% relative humidity (RH). Unbleached sack paper (SP, 75 g/m^2 at 50% RH) made of Nordic softwood kraft (NSK) was obtained from a European paper mill. Laboratory-grade citric acid monohydrate (CA) was obtained from VWR International bvba (Leuven, Belgium), and the technical-grade mDMDHEU (Fixapret AP liq c) was obtained from Archroma (Cal Coracero, Spain). Sodium hypophosphite monohydrate (SHP) for the CA crosslinking was obtained from VWR International bvba (Leuven, Belgium), and the technical-grade catalyst Fixapret Catalyst LF for mDMDHEU crosslinking was obtained from Archroma (Cal Coracero, Spain). Deionized water was used for all dilutions.

Crosslinking

For the chemical crosslinking, the laboratory hand sheet paper (HP) and SP samples were immersed in the aqueous solution containing the crosslinking agent and its catalysts for 60 s at 23 °C. After soaking, the excess liquid flowing on the paper surfaces was removed using blotting paper. The sheets were then wet-pressed and dried in accordance with ISO 5269-1 (2005). Finally, the sheets were cured in an oven at 155 °C for 20 min.

Table 1. HP and SP Crosslinking: Compositions of the Water Soaking Solutions, Wet Pickup of the Solutions after Wet Pressing, and the Paper Weight Gain After Curing the Dried Papers

	Soaking Solutions					
Sample Codes	Citric Acid + SHP	Fixapret AP + Catalyst LF	Wet Pick-up (%)	WPG (%)	Paper Density (kg/m^3)	Contact Angle ($\Theta_{1\,s}$)
HP Ref.			-	-	524 ± 6	NM*
HP CA 20 g/L	20 g/L + 6 g/L		59	2.2	513 ± 10	NM
HP CA 40 g/L	40 g/L +12 g/L		68	3.7	524 ± 7	NM
HP Fix 30 mL/L		30 mL/L + 9 mL/L	63	2.4	529 ± 7	NM
HP Fix 60 mL/L		60 mL/L + 18 mL/L	66	4.0	523 ± 8	NM
Sack Ref.			-	-	759 ± 10	110
Sack CA 20 g/L	20 g/L + 6 g/L		48	2.7	622 ± 12	89
Sack CA 40 g/L	40 g/L + 12 g/L		53	4.4	694 ± 15	69
Sack Fix 30 mL/L		30 mL/L + 9 mL/L	46	3.1	685 ± 18	81
Sack Fix 60 mL/L		60 mL/L + 18 ml/L	48	4.3	691 ± 18	75

REF = Non-crosslinked control sample
NM = Not measurable due to immediate water absorption

Reference papers were tested without further treatments. The chemical dosages, as well as the paper wet pickup after wet pressing, and the paper weight percentage gain (WPG %, RH 50%), after curing, are shown in Table 1. Because of the complexity of quantitative determination of CA and mDMDHEU contents in paper, the effects of the crosslinking on the handsheet water absorption and strength properties are considered as a function of the added amounts of the crosslinking agents in the water soaking solution.

Biodegradation Tests

The paper samples' biodegradation rates were compared using a 48-h enzymatic hydrolysis test and a 2-month soil burial test. The enzymatic hydrolysis test followed the method described by Leppänen *et al*. (2020). In the test, paper samples were hydrolyzed using a mixture of cellulase, mannanase, xylanase, and β-glucosidase enzymes for 48 h at 40 °C and at 25 g/L solids content. An enzyme dose of 50 FPU/g was used. The degree of sample degradation (%) was calculated by comparing the amount of the liberated reducing sugars (g/L) to the initial amount of the sample (g/L). The sample size of the paper used was 100 mg. Two replicates of each sample were measured.

The soil burial test was done using a method in line with EN 13432 (2001). In the test, the paper samples were attached to plastic slide frames (24 mm x 36 mm) and buried in soil to a depth of 5 to 20 cm. The moisture content of the soil was 25% to 30%. The composting box covered by a lid was kept in a dark room at 23 °C. Visual inspection of the samples was done after 2 months. After lifting the slide frames and cleaning the paper samples, the percentage area of formed holes in the paper samples was visually estimated. Three replicates were assessed for each sample. It is good to note that in the soil buried paper samples can become thinner before getting the first holes. However, the evaluation of the results depends only on the total area of the holes created in the paper

Testing of fungus growth on the paper samples was done following TAPPI method T487 cm-93 (1993). In the test, paper samples are placed on mineral salt agar plates followed by uniform inoculation of the samples in a water solution with test fungus spores (*Aspergillus niger*, VTT type culture collection VTT-D-7005). The agar plates were incubated at 25 ± 2 °C for 7 days, followed by visual assessment of the fungal growth on the paper samples. In the test, suppressed fungus growth indicates the action of biocidal agents. All samples, including the references, were tested in triplicate.

Testing of Paper Physical and Mechanical Properties

The HP and SP samples were tested according to ISO standards (Table 2). For sack paper, the strength properties were measured in both the machine and cross directions (MD, CD). The water absorption and drying of the paper stored at 23 °C and RH 50% were measured by immersing 3.0 cm × 3.0 cm paper pieces in water for 10 min, followed by the removal of excess water using blotting paper. The paper pieces were then weighed, and the weight percentage change (WPG) was calculated. The drying rate of the wetted paper pieces was measured by allowing the pieces to dry freely on a plastic net at 23 °C and RH of 50%, and by weighing the samples after 0, 10, 20, and 40 min. Three replicates of each sample were measured. Water contact angle measurements were made using Theta One Attension optical tensiometer (Biolin Scientific, Model C204A, Espoo, Finland). Water drop size used was 5 µL. The reported contact angles are averages of 5 parallel measurements.

Table 2. Testing of Paper Physical and Mechanical Properties

Grammage (g/m^2)	ISO 5270 (2012)
Bulk (kg/m^3)	ISO 534 (2011)
Tensile Index (Nm/g), Strain at break (%),	EN ISO 1924-2 (2008)
Wet Tensile Strength Index (Nm/g)	ISO 3781 (2011)
Tear Index (mNm^2/g)	ISO 1974 (2012)

RESULTS AND DISCUSSION

According to the performed 48-h enzymatic hydrolysis tests and the 2-month soil burial tests, chemical crosslinking is an effective method to make kraft paper more resistant to biodegradation (Fig. 2).

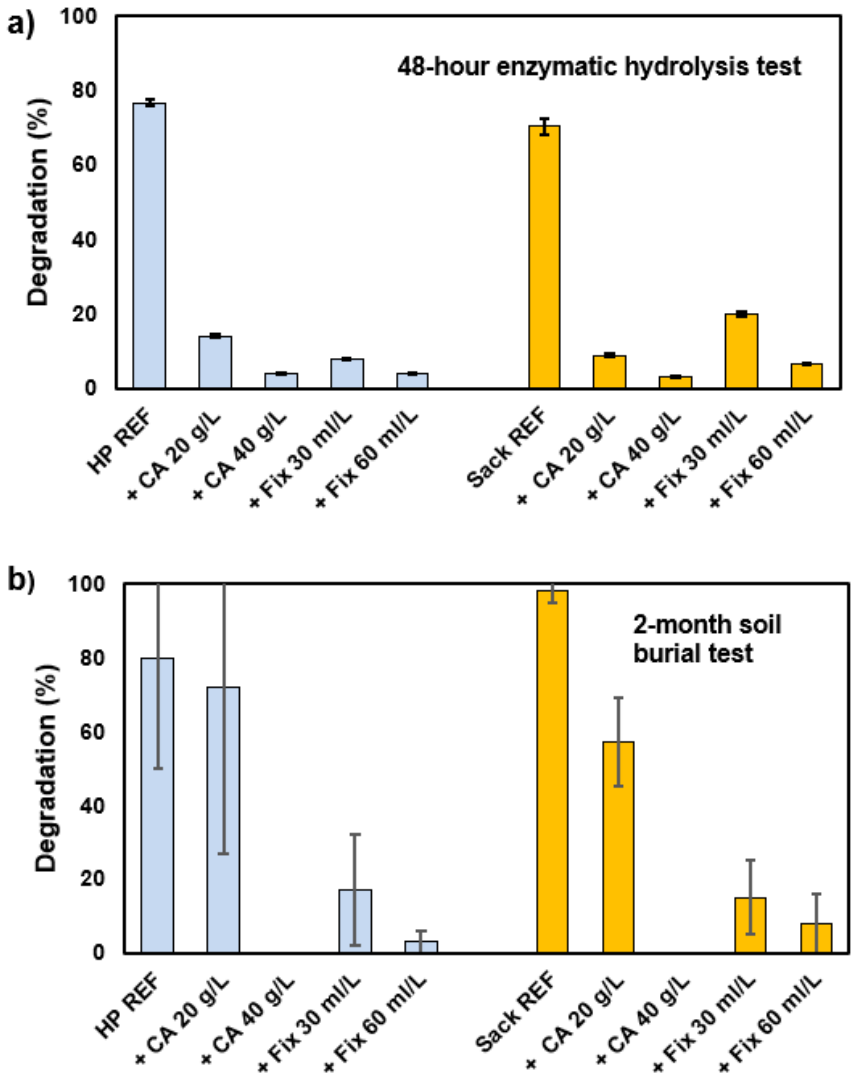

Fig. 2. The effect of chemical crosslinking on the biodegradation in a) a 48-h enzymatic hydrolysis test and b) in a 2-month soil-burial test. The "mL/L" and "g/L" indicate the added amount of the crosslinking agent in the handsheet soaking solution.

The results are in accordance with the observations by Smith *et al*. (2021) regarding the effect of chemical crosslinking with mDMDHEU on the biodegradation rate of cotton fabric in moist soil. Taking into account the WPG of the paper samples, the molecular weights of CA (192 g/mol), mDMDHEU (206 g/mol), and cellulose glucose units (180 g/mol), and supposing that each crosslinking agent molecule substitutes a maximum of two cellulosic hydroxyl groups, the measured paper degradation rates of 3.1% to 6.5% in the enzymatic hydrolysis test were achieved with well below a DS value of 0.1 of the cellulose. In a corresponding 48-h enzymatic hydrolysis test, the biodegradation rate of methylated cellulose was around 5% with a DS value of 1.7 (Leppänen *et al*. 2020). Thus, it seems that compared to various cellulose substituents, chemical crosslinks formed using CA or mDMDHEU are more efficient at reducing the accessibility of the cellulose backbones to the degrading enzymes. The results of the 2-month soil burial test are in line with the results of the 48-h soil burial test (Fig. 2). In the performed fungus resistance test the crosslinked papers did not differ from the reference papers. Therefore, the crosslinked papers had no biocidal effect on the inoculated fungus (*Aspergillus niger*).

According to the water soaking test, chemical crosslinking substantially decreased the paper's liquid water absorption (Fig. 3). The papers also became dry in a shorter time than the reference papers. These results are in line with the observations of the preceding study by Korpela *et al*. (2023). Based on the literature, a decrease in the paper water absorption alone can play a significant role in the biodegradation rate of paper (Andrady *et al*. 1992; Kwon *et al*. 2021; Vikman *et al*. 2002). According to the wet-strength measurements, chemical crosslinking had a noticeable increasing effect on the paper wet strength (Fig. 4). In practice, low water absorption, a short drying time, and a high wet strength can be beneficial technical features in building papers, mulching papers, and exterior packaging papers.

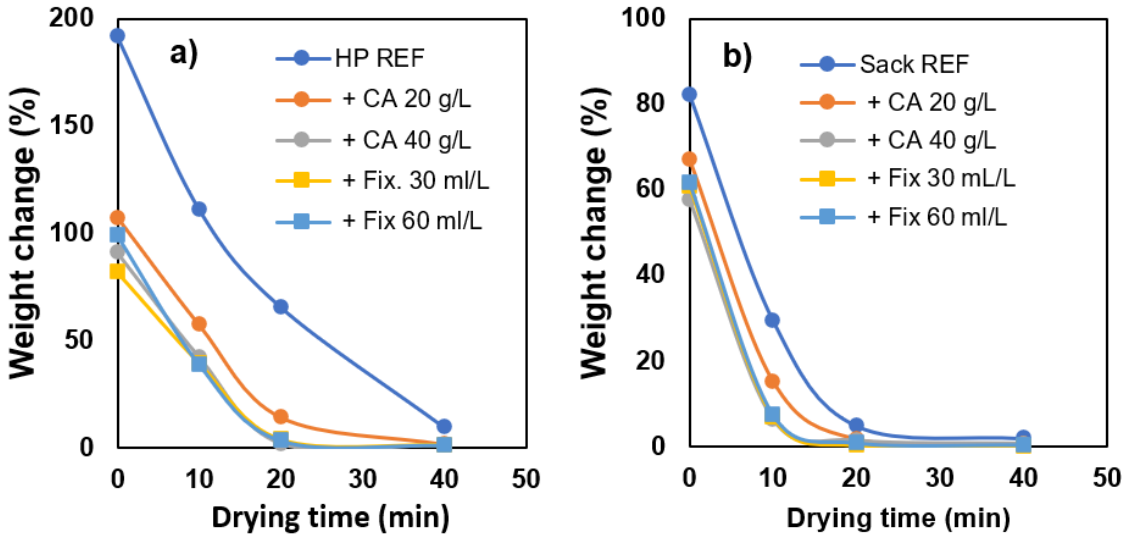

Fig. 3. The weight change (%) of the reference papers and crosslinked papers after soaking (10 min) and subsequent drying (23 °C, RH 50%) of the papers for 0 min, 10 min, 20 min, and 40 min. The "mL/L" and "g/L" indicate the added amount of the crosslinking agent in the handsheet soaking solution.

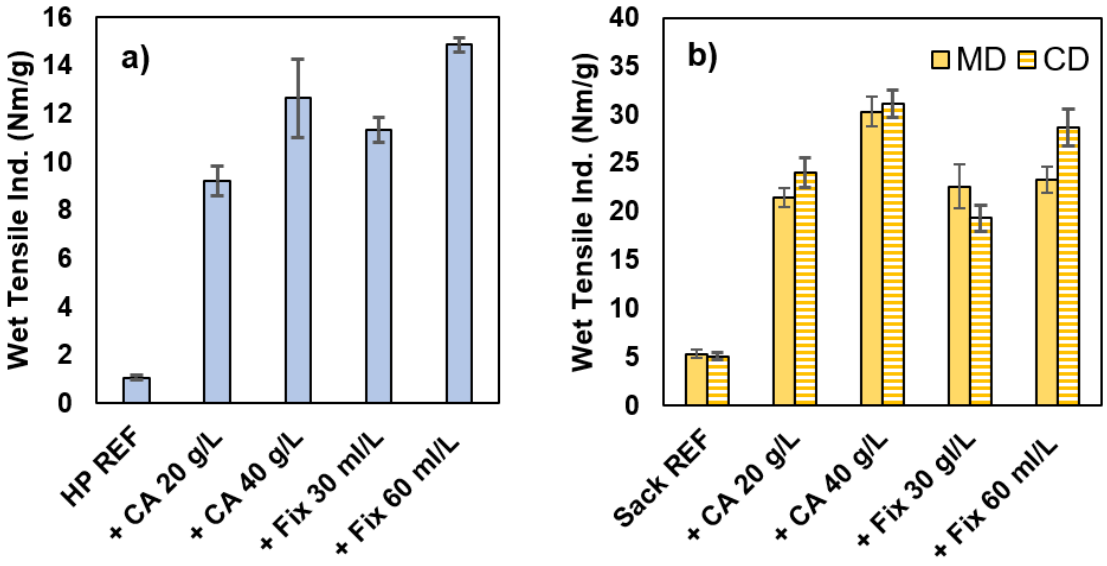

Fig. 4. The effect of chemical crosslinking with CA and mDMDHEU on NBSK handsheets and sack paper wet tensile strength (Nm/g) for 0 min, 10 min, 20 min, and 40 min. The water soaking time was 60 s. The "mL/L" and "g/L" indicate the added amount of the crosslinking agent in the handsheet soaking solution.

Table 3. Effect of Chemical Crosslinking Treatments on the Properties of Tested Papers

Sample Code		Tensile Index (Nm/g)	Elastic Modulus (N/mm^2)	Strain at Break (%)	Tear Index (mNm^2/g)
NBSK Handsheet Paper (100 g/m^2)					
HP Ref.		23.1 ± 0.7	1915 ± 54	2.4 ± 0.2	18.9 ± 0.9
HP CA 20 g/L		26.7 ± 1.3	1862 ± 63	2.1 ± 0.1	11.5 ± 1.1
HP CA 40 g/L		33.7 ± 1.3	2144 ± 100	2.0 ± 0.2	6.9 ± 0.4
HP Fix 30 mL/L		27.6 ± 0.6	1807 ± 26	2.0 ± 0.2	8.4 ± 0.2
HP Fix 60 mL/L		30.0 ± 1.7	1812 ± 90	1.9 ± 0.2	6.0 ± 0.3
NSK Sack Paper (75 g/m^2)					
Sack Ref.	MD	64.3 ± 3.3	2880 ± 236	8.1 ± 0.5	11.3 ± 0.6
	CD	61.3 ± 4.8	3340 ± 142	6.8 ± 0.6	12.7 ± 0.7
Sack CA 20 g/L	MD	49.0 ± 5.0	1726 ± 236	6.5 ± 0.8	5.7 ± 0.2
	CD	52.3 ± 3.1	2574 ± 208	4.2 ± 0.5	6.6 ± 0.1
Sack CA 40 g/L	MD	47.4 ± 2.8	2072 ± 399	4.5 ± 0.5	3.7 ± 0.3
	CD	49.9 ± 3.7	2540 ± 391	3.4 ± 0.3	4.3 ± 0.3
Sack Fix 30 mL/L	MD	64.9 ± 4.4	1950 ± 181	8.4 ± 0.3	8.2 ± 0.9
	CD	55.4 ± 6.6	2283 ± 258	6.0 ± 0.9	10.1 ± 0.9
Sack Fix 60 mL/L	MD	60.8 ± 3.7	2227 ± 257	7.4 ± 1.0	6.5 ± 0.2
	CD	55.1 ± 7.7	2587 ± 236	4.6 ± 1.2	7.3 ± 0.8

Ref = Non-crosslinked control sample

Table 3 shows the effect of chemical crosslinking treatments on the dry strength properties of the tested papers. Overall, with the exception of the substantially increased tensile strength of the handsheets, the observed effects are in line with earlier published results by Luner *et al.* (1993) Korpela *et al*. (2023). The reason for the increased tensile

strength is not clear, but it is most likely to be caused by the formation of inter-fiber crosslinks between the unrefined NBSK fibers. The reduction in the paper strain at break and tear strength indicate a crosslink-induced reduction in the paper formability.

The results of the present study indicate that chemical crosslinking could be used as a non-biocidal tool to make paper more resistant to biodegradation. Because of the increase in the paper wet strength, balancing paper biodegradability, and recyclability may be needed in some cases. In applications where the paper tear strength or folding endurance are critical properties, it is worth testing whether they can be maintained at an adequate level by adjusting the pulp refining or using strengthening agents, which also influence the paper tear strength and flexibility.

In the present study, handsheet paper and sack paper samples were immersed in an aqueous solution of crosslinking agent to achieve an even and repeatable absorption of crosslinking agent in the paper samples. In paper manufacturing, a more viable option for paper immersion could be the use of a size press. Perhaps the most challenging step in the treatment to implement in paper mills is the curing stage. In pad-dry finishing of cotton fabrics with mDMDHEU, the usual curing time is 3 to 5 min and the curing temperature is 140 to 155 °C (Shindler and Hauser 2004; Dehabadi *et al*. 2013; Choudhury 2017). Therefore, for the curing of the crosslinking agent a separate curing treatment is probably needed. From the point of view of the viability of paper crosslinking treatment, it would be desirable to find solutions to shorten the required curing time and to lower the curing temperature.

CONCLUSIONS

1. Chemical crosslinking with either citric acid (CA) or 1,3-dimethyl-4,5-dihydroxyethyene urea (mDMDHEU) made kraft paper more resistant to biodegradation. This was demonstrated by both enzymatic hydrolysis and soil burial tests.
2. The improved resistance to biodegradation was not due to a biocidal effect but due to decreased accessibility of the fibers to water and degrading enzymes. Partial substitution of cellulosic hydroxyl groups by the crosslinks may also impede the action of the cellulolytic substrate-specific enzymes.
3. The results of the present study indicate that chemical crosslinking could be used as a non-biocidal tool to make paper more resistant to biodegradation. The treatment also gives the paper substantially more wet strength. A downside of crosslinking treatment is a reduction in the paper tear strength. For the curing of either CA or mDMDHEU, a relatively high curing temperature and time is needed.

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