A Comparative Study of the Effects of Chemical Crosslinking Agents on NBSK Handsheet Properties

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Chemical crosslinking is an established method for improving the wet performance of paper. In the chemical crosslinking process, covalent bonds are formed between cellulosic surfaces. The formed intra- and inter-fiber bonds increase the paper's wet strength and reduce its water absorptivity. The majority of published studies concern crosslinking treatments with glyoxal, citric acid (CA), or with 1,2,3,4-butanetetra-carboxylic acid (BTCA). The most severe disadvantage of the crosslinking treatments with glyoxal, CA, and BTCA is that the formed crosslinks make the fibers and the paper more brittle. This downside effect has largely impeded the utilization of crosslinking in paper and paperboard making. In the present study, handsheets made from Nordic bleached softwood kraft pulp (NBSK) were crosslinked with methylated 1,3-dimethylol-4,5-dihydroxyethylene urea (mDMDHEU), which is commonly used in cotton fabric finishing. Similar to using glyoxal and citric acid, crosslinking with mDMDHEU notably increased the handsheet wet strength and decreased the water absorption. Compared to the use of glyoxal or CA, the crosslinking with mDMDHEU did not make the handsheets that brittle. These results suggest that mDMDHEU could be a more viable crosslinking agent for improving the wet performance of paper products.

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INTRODUCTION

Crosslinking cellulosic material *via* covalent bonds in chemical crosslinks prevents the mutual movement and relocation of adjacent cellulosic chains and fibers as the material is being wetted, dried, and mechanically stressed. Chemical crosslinking is commonly used for cotton fabric finishing. In the so called 'pad-dry-cure' technique, the fabric is first soaked in a crosslinking agent aqueous solution. This is followed by squeezing out any extra liquor, smoothening and drying of the fabric, and curing the crosslinking agent. The crosslinking reactions take place mostly during the curing stage, at elevated temperatures. Some catalyst is usually added to reduce the required time and temperature for complete curing. The formed chemical crosslinks allow for retention of the fabric smoothness and reduce the shrinking of cotton fabrics in washing and drying, as well as during the later use of the fabric. Because of these effects, the crosslinking treatments are also called "durable-press" or "easy-care" treatments. In cotton fabric crosslinking, the most widely used crosslinking agents are 1,3-dimethylol-4,5-dihydroxyethylene urea (DMDHEU) and its partially methylated 'low formaldehyde' or 'very low formaldehyde' versions ("mDMDHEU"). These achieve the desired finishing effects at a relatively low cost,

compared to some other crosslinking agents, such as glyoxal, glutaraldehyde, and polycarboxylic acids, with less harmful effect on the fabric strength, color, and other relevant fabric properties. Crosslinking of cellulose with glyoxal, CA, and with mDMDHEU with is shown in Fig. 1. (Shindler and Hauser 2004; Dehabadi *et al.* 2013; Mukthy *et al.* 2014; Choudhury 2017).

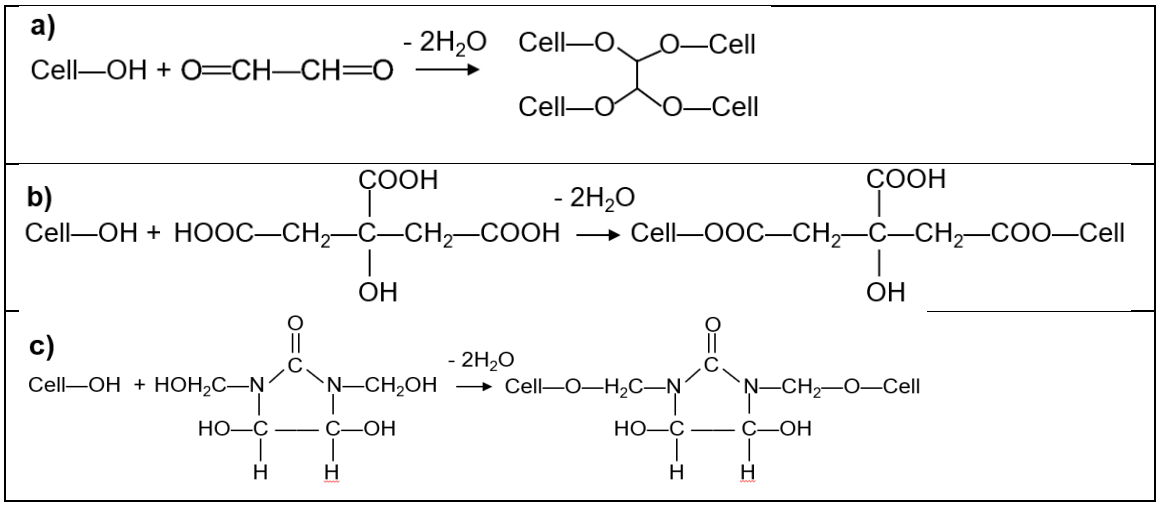

Fig. 1. Crosslinking of cellulose with a) glyoxal, b) CA, and c) mDMDHEU

Some earlier published studies indicate that the paper wet strength and dimensional stability can be substantially improved by chemical crosslinking treatments. The crosslinking agents used for this purpose were formaldehyde (Stamm 1959; Caulfield and Weatherwax 1976), glyoxal (Eldred and Spicer 1963), glyoxal and glutaraldehyde (Xu *et al.* 2002), citric acid (CA), and 1,2,3,4-butanetetracarboxylic acid (BTCA) (Caulfield 1994; Horie and Biermann 1994; Yang and Xu 1998; Widsten *et al.* 2014). The studies showed that chemical crosslinking results notably increased paper wet strength. In a study by Caulfield (1994), the crosslinking of paperboard made from bleached kraft, furnished with BTCA, reduced the creep deformations of the boards in cyclic humidity. The CA-treated corrugated boxes showed a greater than three-fold increase in their resistance to compressive creep.

The most severe reported disadvantage of paper crosslinking treatments is embrittlement of the paper, resulting in greatly decreased folding endurance and strain during the breakage of the paper (Caulfield 1994; Horie and Biermann 1994; Yang and Xu 1998; Korpela and Orelma 2020). The embrittlement is believed to be a consequence of reduced relative flexibility of the cellulose chains and, thus, reduced formability of the fibers, and paper, by the formed crosslinks. Caulfield (1994) pointed out that although the wet performance of paper is greatly improved by chemical crosslinking, it is rare in practice that the importance of the wet properties override the dry performance of the paper. It is worth mentioning that the manufacture of paper from chemically pre-crosslinked chemical pulp fibers has resulted in improved fiber and paper web dewatering properties. This has also increased paper bulk but has substantially decreased the paper strength properties (Westervelt and Elston 1995; Korpela and Tanaka 2015).

The idea of the present study was to examine the effects of mDMDHEU crosslinking on the mechanical and water sorption properties of Nordic bleached softwood

kraft (NBSK) handsheets, compared with the effects of glyoxal and CA crosslinking. The performed crosslinking treatments were followed the pad-dry-cure method. To withstand the soaking stage, the used handsheets were wet-strengthened with a wet-strength agent, polyamide-amine-epichlorohydrin (PAE). According to general understanding, the PAE increases paper wet strength by forming self-crosslinked, fiber-bond networks protecting fiber-to-fiber bonds when paper is wetted by water (Häggkvist *et al.* 1998; Ozaki *et al.* 2006; Siqueira 2012). The treatments were completed using appropriate crosslinking reaction catalysts and curing conditions, for the crosslinking agents. The desire behind this experimental study was to find out whether crosslinking with the mDMDHEU, which shows good overall performance for cotton fabric crosslinking, would improve handsheet wet performance without severe handsheet embrittlement. This could open up new possibilities for the utilization of chemical crosslinking in the manufacture of water and moisture tolerant paper products. According to the authors' knowledge, no studies regarding chemical crosslinking of paper or paperboard with mDMDHEU have been previously published.

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EXPERIMENTAL

Materials

The NBSK pulp sheets were obtained from a Finnish pulp mill. The polyamide-amine-epichlorohydrin (PAE) was a technical-grade product FennoSize PA21 (Kemira OyJ, Espoo, Finland) used industrially for the wet-strengthening of paper products. The technical-grade glyoxal (OCHCHO) was obtained as a 40% water solution from Thermo Fisher GmbH (Kandel, Germany) and laboratory-grade citric acid monohydrate from VWR International bvba (Leuven, Belgium) were used as received. The mDMDHEU (Fixapret AP liq c) was a technical-grade product provided by Archroma (Cal Coracero, Spain). The crosslinking catalyst alum, aluminium sulphate tetradecahydrate employed for glyoxal crosslinking, was obtained from Kemira OyJ (Espoo, Finland). Sodium hypophosphite monohydrate 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). All chemicals were used as such in the laboratory trials. De-ionized water was used for all dilutions.

Preparation of Laboratory Sheets

Laboratory paper sheets were made using uncirculated ion-exchanged water following ISO 5269-1 (2005). Before the handsheet making, the NBSK-pulp sheets were dispersed in water and refined to 19.0 °SR-value using a Voith LR1 laboratory refiner (Voith AG, Heidenheim, Germany). The °SR-value was measured according to EN ISO 5267-1 (1999). The pH of the pulp was adjusted to 6.5-7.0 using 1 M HCl and 1 M NaOH solutions. A diluted PAE water solution (0.2 wt%) was added to the pulp suspension. The added amount of PAE was 0.3 wt%, on a dry fiber basis. The pulp suspension was mixed for 10 s after the PAE addition, followed by immediate drainage of the suspension. For curing of the PAE, the dried laboratory paper sheets were oven-heated at 80 °C, for 120 min. The targeted handsheet grammage was 80 g/m^2 with a RH of 50%.

Crosslinking

For the chemical crosslinking, the handsheets were immersed in the aqueous crosslinking agent solutions containing the catalysts for about 30 s (20 °C). After soaking, the excess liquid flowing on the sheet surfaces was removed using a 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 elevated temperature. The chemical dosages, wet pickup of the handsheets, after the wet pressing, and the curing conditions are shown in Table 1. Because of the difficulty in the quantitative analysis of glyoxal, CA, and mDMDHEU 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.

Table 1. Handsheet Crosslinking: Composition of the Water Soaking Solutions, Wet Pickup of the Solutions after Handsheet Wet Pressing and Curing Conditions of Wet Pressed and Dried Handsheets.

Sample Code	Crosslinker	Catalyst	Wet Pickup	Curing
REF	-	-	-	-
Glyoxal 1	Glyoxal 20 mL/L	Alum 6 g/L	38 wt%	135 °C, 10 min
Glyoxal 2	Glyoxal 40 mL/L	Alum 12 g/L	40 wt%	135 °C, 10 min
Glyoxal 3	Glyoxal 60 mL/L	Alum 18 g/L	41 wt%	135 °C, 10 min
CA 1	CA 15 g/L	SHP 5 g/L	40 wt%	155 °C, 20 min
CA 2	CA 30 g/L	SHP 9 g/L	42 wt%	155 °C, 20 min
CA 3	CA 60 g/L	SHP 18 g/L	39 wt%	155 °C, 20 min
mDMDHEU 1	Fixapret AP 20 ml/L	Catalyst LF 6 ml/L	39 wt%	150 °C, 15 min
mDMDHEU 2	Fixapret AP 40 ml/L	Catalyst LF 12 ml/L	40 wt%	150 °C, 15 min
mDMDHEU 2	Fixapret AP 60 ml/L	Catalyst LF 18 ml/L	40 wt%	150 °C, 15 min

REF = Non-crosslinked control sample.

Testing of Laboratory Paper Sheets

The laboratory handsheets were tested according to the ISO and TAPPI standards (Table 2). The liquid water absorption and drying of the handsheets stored at 23 °C and relative humidity (RH) 50% were measured by immersing 2.0 cm × 2.0 cm handsheet pieces stored in water for 10 min followed by the removal of excess water using blotting paper. The pieces were then weighed and the percentage weight change (%) was calculated. The drying rate of the wetted handsheet pieces was measured by allowing the pieces to dry freely on plastic net at 23 °C and a RH of 50% - by weighting the samples after 0, 10, 20, and 40 min. The reported results are averages of 3 parallel measurements.

The water vapor absorption and desorption were determined using a dynamic vapour sorption (DVS) Resolution gravimetric vapor sorption instrument (Surface Measurement Systems, London, UK). Approximately 10 mg of handsheet was placed in the microbalance of the DVS system, and the sample was first kept at 25 °C, at a RH of 50% with nitrogen gas (flow rate 200 mL/min) until the mass change was lower than 0.002 mg/min over a 10 min period. The RH in the chamber was adjusted stepwise to 90, 50, 30, and 50% while keeping the temperature at 25 °C. In each step, the RH was kept constant until the mass of the handsheet sample reached an equilibrium (mass change lower than 0.002 mg/min over a 10 min period). A full sorption cycle, including adsorption and

desorption, was performed for each sample. Two parallel crosslinked handsheet samples were tested and the average values are reported as the percentage weight change (%). For the reference sample, three parallel measurements were performed.

Table 2. Utilized Handsheet Test Methods

Grammage (g/m^2)	ISO 5270 (2012)
Bulk (kg/m^3)	ISO 534 (2011)
Tensile Index (Nm/g), Strain at Break (%), Tensile Energy Absorption (J/m^2)	EN ISO 1924-2 (2008)
Wet Tensile Strength (kN/m)	ISO 3781 (2011)
Tear Index (mNm^2/g)	ISO 1974 (2012)
Resistance to Bending, Bending Length 10 mm, Bending Angle 15° (mN)	ISO 2493-1 (2010)
Internal Bonding Strength, Scott-Bond (J/m^2)	ISO 16260 (2016)
Double Folding Number, Schopper	ISO 5626 (1993)

RESULTS AND DISCUSSION

Figure 2 shows the effect of the contents of glyoxal, CA, and mDMDHEU crosslinking agents on the handsheet weight percentage gain (WG%) after 10 min water soaking followed by subsequent drying for 10, 20, and 40 min, at 50% RH. Before soaking, the samples were stored at 50% RH ('0 min sample'). In all cases, chemical crosslinking resulted in a decreased WG%. This effect is in line with the observed effects of chemical crosslinking on cotton fabric water sorption properties (Shindler and Hauser 2004; Dehabadi *et al*. 2013; Choudhury 2017). In an earlier study by Korpela and Tanaka (2015), with chemical crosslinking of bleached hardwood kraft fibers using mDMDHEU, the water retention value (WRV) was reduced by 25%. It is obvious that the chemical crosslinking treatments decreased the fibers' capability to swell and absorb liquid water. According to the obtained results (Fig. 2), the crosslinking treatments did not affect the drying rate, *i.e.*, the weight of water evaporation per unit area per unit time, but the drying time to a certain moisture content depends on the amount of water originally held by the handsheets. It is worth mentioning that this is in accordance with the rate of drying of various fabrics (Fourt *et al.* 1951). For packaging papers, and some specialty papers, such as building papers, lower water uptake and faster drying of the paper, to a certain moisture content, may be advantageous - as it may lower the risk of both wetting induced creep and the breaking of the packaging. It may also lower the risk of mold growth in the paper.

The effect of the chemical crosslinking treatments on the handsheet water vapor absorption and desorption, at various RH values, is shown in Fig. 3. The samples were first kept at 25 °C, at a RH 50%, until constant moisture content was achieved. According to the results, the chemical crosslinking treatments slightly reduced both the water absorption and desorption. This is likely due to a reduction in the accessibility of hydrophilic surfaces of the fibers due to crosslinks limiting the swelling between fibrils/fibres. However, the crosslinking treatments did not have any notable effect on the water contact angles (Fig. 4). It is worth mentioning that for cotton fabrics, low moisture absorption is not necessarily a desired property, as it may worsen the comfort of the fabric in use (Wallace 2002; Motlogelwa 2018).

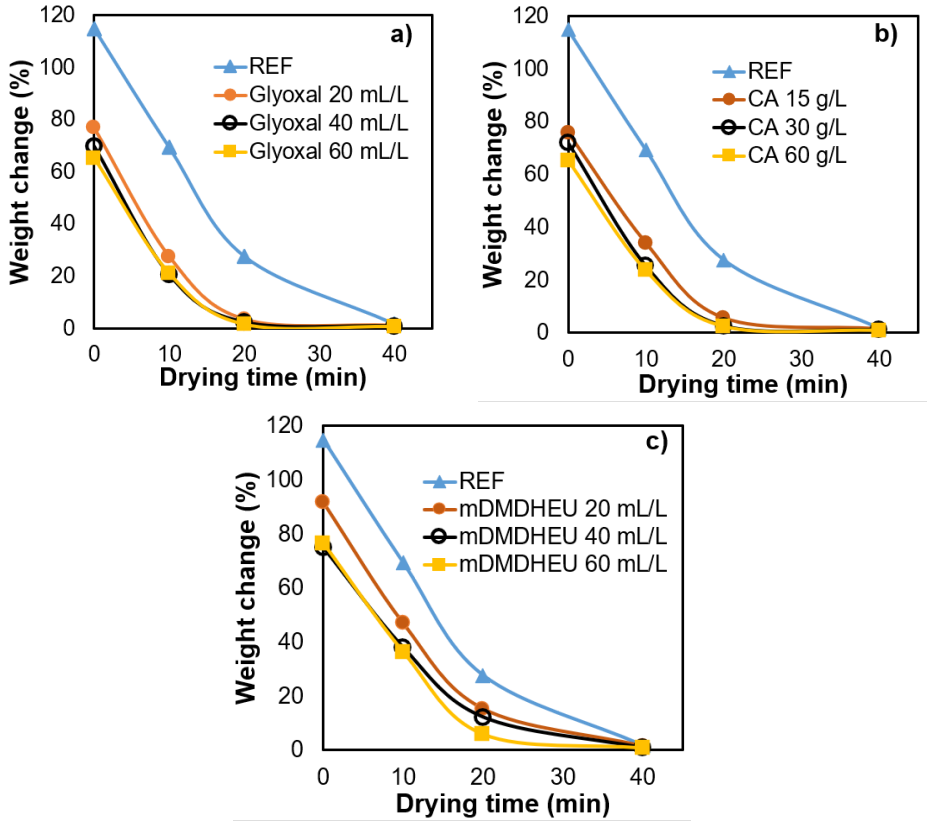

Fig. 2. Weight change (%) of the reference handsheets and crosslinked handsheets after water soaking (10 min) and subsequent drying (23 °C, RH 50%) of the samples. The handsheets were stored at a RH of 50%, at 23 °C before the soaking stage. The weight change (%) was immediately determined after water immersion ('0 min' sample) and after subsequent drying for 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 water solutions.

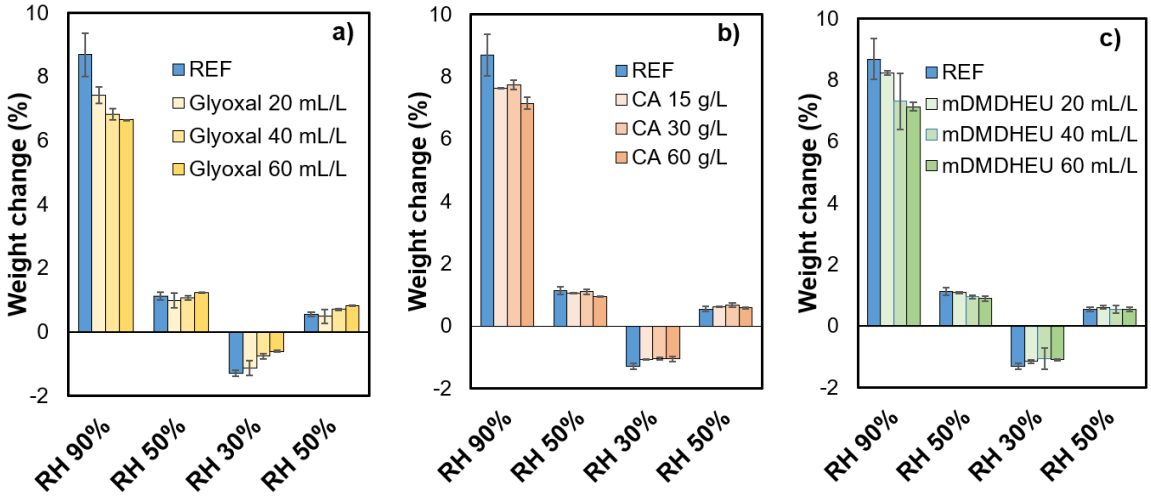

Fig. 3. Effect of varying the RH on the water absorption and desorption of a) glyoxal-, b) CA-, and c) mDMDHEU-crosslinked handsheets. After keeping the samples at 25 °C and at an RH of 50%, the RH was adjusted stepwise to 90%, 50%, 30%, and 50%. The "mL/L" and "g/L" indicate the added amount of the crosslinking agent in the handsheet soaking water solutions.

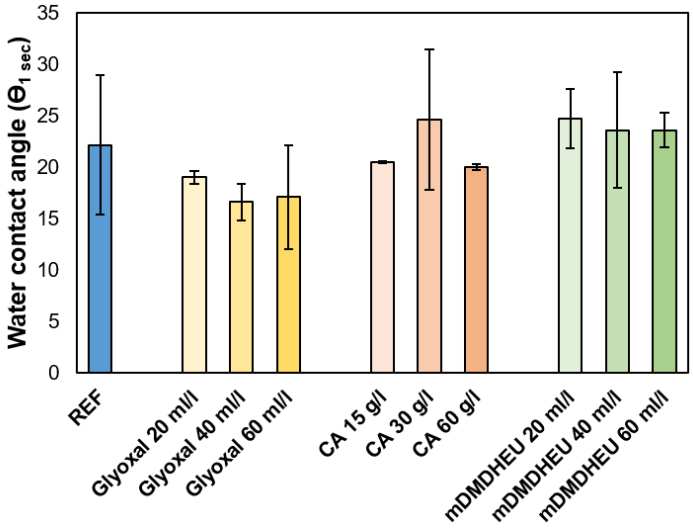

Fig. 4. The effect of chemical crosslinking with glyoxal, CA, and mDMDHEU, on the water contact angle ($\theta_{1\,s}$) measured on the handsheets, wire side. The "mL/L" and "g/L" indicate the added amount of the crosslinking agent in the handsheet soaking water solutions.

Figure 5 shows the effect of chemical crosslinking with glyoxal, CA, and mDMDHEU on the dry strength properties of the NBSK handsheets. According to the results, crosslinking treatments with mDMDHEU resulted in a smaller decrease in the hand-sheet strain at break, as well as in the tensile energy absorption (TEA), double folding number, and tear strength, compared to the crosslinking with glyoxal and CA. Thus, the crosslinking with mDMDHEU has less detrimental effects on the handsheet formability and toughness, compared with crosslinking using glyoxal or CA. The handsheet embrittlement, due to glyoxal and CA crosslinking, was in accordance with earlier published results (Caulfield 1994; Horie and Biermann 1994; Yang and Xu 1998; Korpela and Orelma 2020).

The effect of the chemical crosslinking treatments on the density, dry tensile strength, z-directional tensile strength (Scott-Bond), and resistance to bending are shown in Fig. 6. The slight increase in the handsheet densities may be a consequence of the 'additional' wet pressing, included in the crosslinking treatments. Further, the retention of the crosslinking agents in the handsheets may affect the handsheet density to some extent. Glyoxal crosslinking resulted in decreased handsheet tensile strength and Scott-bond. The actual reason for this is unfortunately not clear and it would necessitate further examinations. The increase of the handsheet Scott-bond due to CA crosslinking may result from the formation of inter-fiber crosslinks. The handsheet wet strength was measured after 1 min and 1000 min (16 h) of water soaking. The chemical crosslinking treatments with the tested crosslinking agents increased the wet tensile strength notably (Fig. 7).

The results of the present study indicate that the chemical crosslinking of paper with a small amount of mDMDHEU could be used for a notable improvement of the paper wet performance, without severe deterioration of the paper formability and toughness, which are characteristic disadvantages of glyoxal and CA crosslinking of paper. Unfortunately, the molecular-level mechanisms behind the different responses of the tested crosslinking treatments are not clear yet. The differences may be related to the different degrees of reduction of the mutual mobility of the cellulosic fibres by the formation of

crosslinks. Or, possibly the effects were due to partial hydrolysis of cellulosic chains occurring in the crosslinking treatments (Wei and Yang 1999; Schindler and Hauser 2004; Harifi and Montazer 2012).

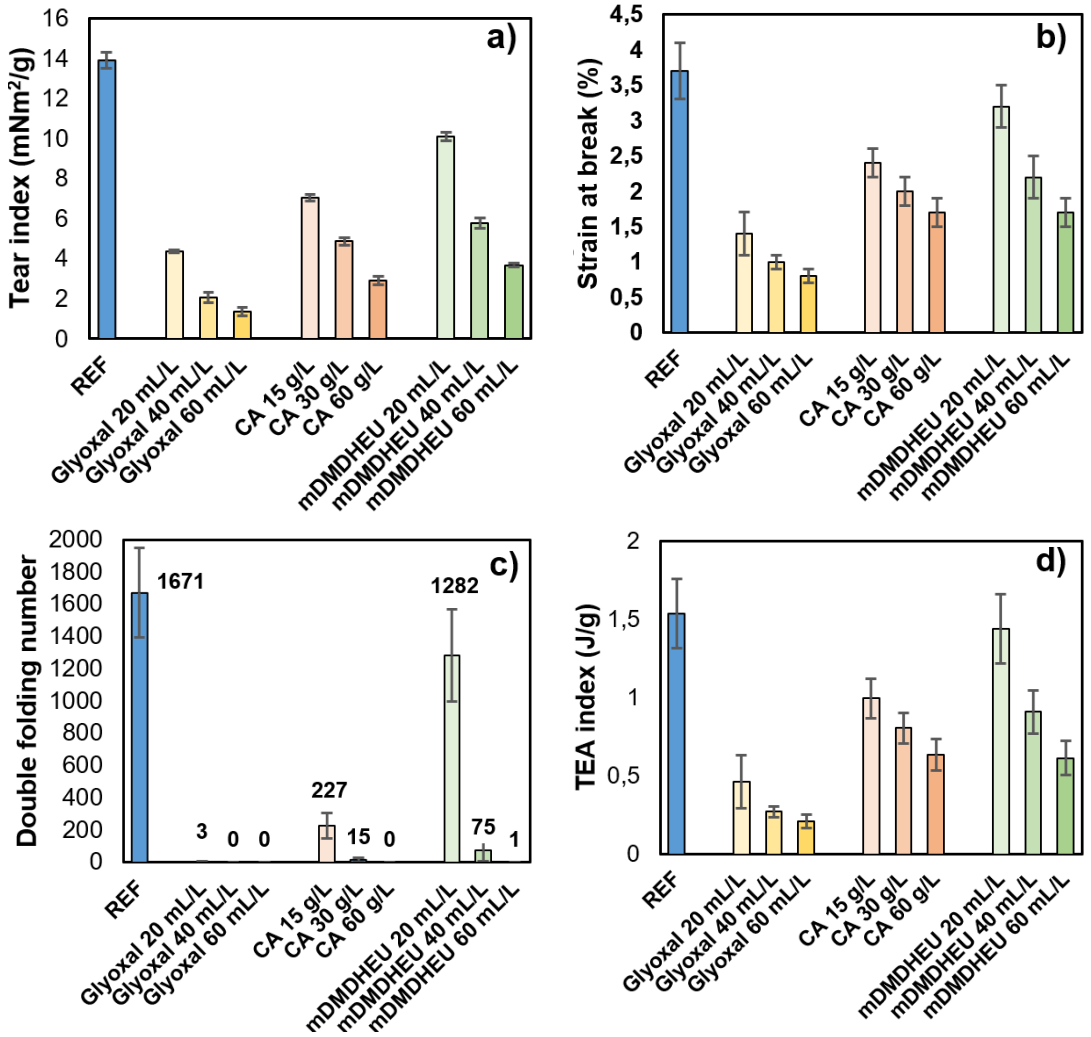

Fig. 5. The effect of chemical crosslinking treatments with glyoxal, CA, and mDMDHEU on an NBSK handsheet: a) TEA index (J/g), b) strain at break (%), c) tear index (mNm^2/g), and double folding number (Schopper). The "mL/L" and "g/L" indicate the added amount of the crosslinking agent in the handsheet soaking water solution.

Conceivable applications for paper chemical crosslinking with mDMDHEU could be filter papers, wallpapers, building papers, laminating papers, and wet strength packaging papers, all making use of the high wet strength, low water absorption, and decent dry toughness of the paper. In development work, it is good to note that use of unnecessarily harsh curing conditions may result in hornification and thermal degradation of the papermaking fibres, and thus deterioration of some relevant properties of the crosslinking treated paper product. Although the amount of free formaldehyde in the tested technical grade mDMDHEU is, according to the manufacturer, "very low," it might still be an issue in papermaking and converting. Testing the performance of "totally formaldehyde free DMDHEU substitutes", which have been developed for cotton textile finishing, will be a subject of further studies.

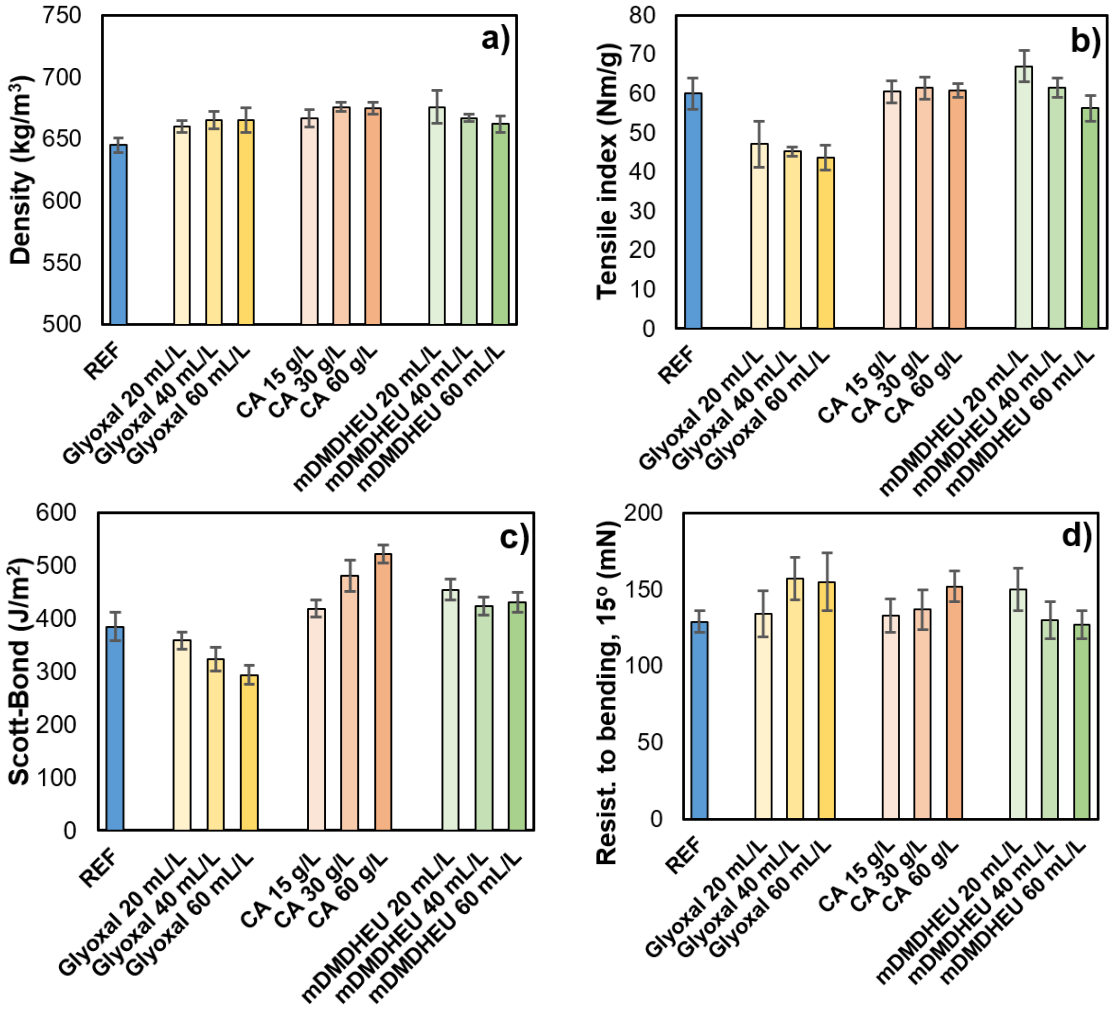

Fig. 6. The effect of chemical crosslinking treatments with glyoxal, CA, and mDMDHEU on an NBSK handsheet: a) density (kg/m^3), b) tensile index (Nm/g), c) Scott-bond (J/m^2), and d) resistance to bending (mN). The "mL/L" and "g/L" indicate the added amount of the crosslinking agent in the handsheet soaking water solutions.

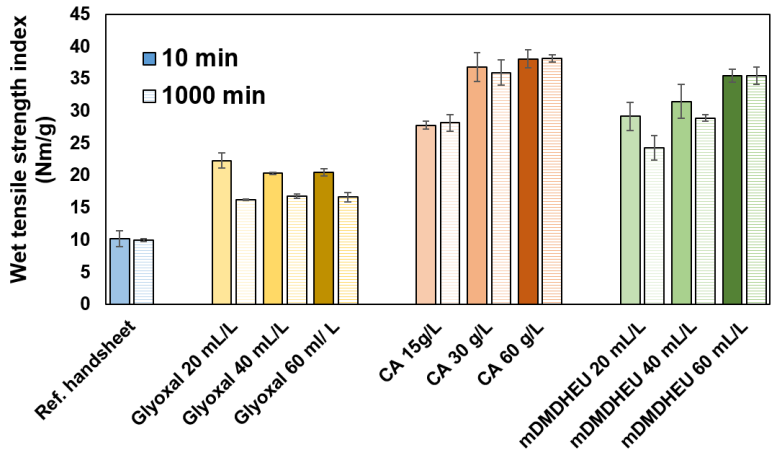

Fig. 7. The effect of chemical crosslinking treatments with glyoxal, CA, and mDMDHEU on NBSK handsheet wet tensile strength (Nm/g). The measurements were taken after 10 min, and 16 h water soaking. The "mL/L" and "g/L" indicate the added amount of the crosslinking agent in the handsheet soaking water solutions.

CONCLUSIONS

1. Similar to chemical crosslinking of Nordic bleached softwood kraft (NBSK) handsheets with glyoxal, and citric acid (CA), chemical crosslinking with the common cotton fabric crosslinking agent methylated 1,3-dimethylol-4,5-dihydroxyethylene urea (mDMDHEU) (Fixapret AP) gave the handsheets a high wet strength, and decreased their water absorption in wet conditions, as well as in highly humid air.
2. Compared to chemical crosslinking with glyoxal or with CA, the crosslinking with mDMDHEU exhibited less detrimental effects on the handsheet folding endurance, tear strength, strain at break, and tensile energy absorption. Chemical crosslinking with mDMDHEU had no noticeable adverse effect on the handsheet dry tensile strength, Scott-bond, or bending resistance.
3. The results suggest that mDMDHEU could be a more viable crosslinking agent for paper crosslinking. Potential applications include various packaging and specialty papers.
4. According to the chemical supplier, the mDMDHEU (Fixapret AP) reagent used contains an extremely small amount of unbound formaldehyde. In terms of paper product applications, the testing performance of totally formaldehyde-free crosslinking agents used for cotton fabric finishing is an interesting subject for further mechanistic and application studies.

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