Cationization of Lignocellulose as a Means to Enhance Paper Strength Properties

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Chemical modification by attaching functional groups to lignocellulosic pulp fibers might be a strategy for improving the pulp, and thereby, paper properties. Several studies have described positive effects on paper strength properties for handsheets prepared from cationic-modified pulp or pulp fractions. This study addressed whether these effects are related to the cationic groups, *e.g.*, by increasing electrostatic attraction and thus paper strength, or rather side effects of the chemical modification process, *e.g.*, fiber flexibilization. To eliminate or at least minimize these side effects, only the fines fraction, which was already highly flexible, was cationized. While the addition of cationized fines affected various pulp and paper properties, most notably drainage time, no difference in strength properties was observed when comparing the addition of cationic or unmodified fines to different pulps or fractions thereof.

Keywords: Chemical modification; Cationization; Pulp; Fines; Paper properties

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INTRODUCTION

While the requirements for many paper properties (*e.g.*, smoothness, bulk, and porosity) can vary depending on the application, high paper strength is a focus for almost all paper grades. A certain level of strength is important for good runnability in the paper machine and in further converting steps, as well as for the end use of the given product. For many paper products, the issue might not be enhancing strength, but rather keeping the required strength properties while reducing raw material costs by reducing basis weight, increasing the content of mineral fillers, or using less expensive pulps. Thus, paper strength is a topic of many studies applying different strategies, such as physical modification (*e.g.*, refining and fractionation), enzyme treatment, and the addition of chemical additives in the wet end (so-called wet and dry strength agents), as well as in surface sizing. Chemical modification of the pulp or of fractions thereof, such as TEMPO (2,2,6,6-tetramethylpiperidinyloxyl) oxidation, grafting fibers with carboxymethylcellulose (CMC), and cationization are also means to improve strength properties (Prado and Matulewicz 2014; Zhao *et al.* 2016), but they are rarely applied in industrial practice.

Cationization of pulp refers to the attachment of cationic functionalities onto lignocellulosic fibers. Similar to the effects of soluble cationic polyelectrolytes, cationization of lignocellulose leads to electrostatic interactions in the suspension, which are apparent during the forming process as improved retention (Käufer and Krause 1984a). In addition to hydrogen bonding, Coulomb, London dispersion, and Van der Waals forces

are responsible for interfiber bonding (Lindström *et al.* 2005; Hirn and Schennach 2015). Cationization may also increase strength properties through its impact on these effects.

Several scientific studies have dealt with the cationization of lignocellulosic pulps or fractions thereof. Krause *et al.* (1981) cationized bleached sulfite pulp. Handsheets of the unrefined pulp showed an increase in breaking length, with a higher degree of substitution (DS), *i.e.*, a higher cationic charge content (Käufer and Krause 1984b). Refining this cationized sulfite pulp in a Jokro mill showed a lower beating resistance, indicated by the lower drainability expressed in Schopper Riegler (SR value) compared with the unmodified pulp after a given treatment. Breaking length was improved for the cationized pulp compared with the unmodified pulp at the same energy input in refining, as well as at the same SR value (Käufer and Krause 1984b). The lower beating resistance was explained by a "spacer effect" (Gruber 2002) introduced with the cationic charges. Cationic groups are chemically bound to the cellulose molecules not only on the surface, but also in the bulk. It is not possible to form intermolecular hydrogen bonds in close proximity to charged groups because of steric hindrance. Thus, the cationic group acts as a spacer between cellulose chains.

In a study on cationized thermo mechanical pulp (TMP), the increase in breaking length was reported to be 24%, and internal bond strength (Scott bond) increased by 60% (Montplaisir *et al.* 2006). In another study, only the long-fiber fraction of the TMP pulp was cationized. Tensile index increased only up to a certain level of cationization, at which point it decreased, although apparent sheet density further increased. The decrease in breaking length was explained by the degradation of cellulose chains at very high cationic charge densities (Ma *et al.* 2010). Contrary to the effects reported for pulps or long-fiber fractions, a recent study comparing the addition of cationized microfibrillated cellulose (MFC) and unmodified MFC as papermaking additives found no significant difference in tensile index for either material (Gao *et al.* 2016).

Most studies have provided no explanation for the positive effect of cationized pulp, fibers, and fiber fractions on strength properties. Gruber (2002) mentioned two possible mechanisms for the improvement. In the first mechanism, cationic groups act as a spacer in the fiber wall, thereby making the fiber more flexible. Thus, conformability and sheet consolidation are improved. In the second mechanism, electrostatic interactions may bring fibers into closer contact and thus, enhance strength properties. Increased fiber flexibility, along with the possibly related increased swelling ability, can also be introduced by other means, such as refining, TEMPO oxidation, or surface grafting with CMC (Zhao *et al.* 2016), and thus, is not exclusively related to cationic groups.

Bringing fibers into closer contact because of cationic-anionic charge interactions is a mechanism directly related to the cationic functional groups, and the results will vary depending on whether soluble polymers are added to the fiber suspension or the cellulosic fibers themselves are cationized. Soluble cationic polymers linking the cellulose fibers may increase the distance between these fibers, while cationic groups directly attached to the cellulose might decrease the distance and bring the molecules into closer contact.

Whether Coulomb forces have an influence on interfiber bonding has not been investigated for cationic pulp fibers, but it has been investigated for soluble dry strength agents. Zhang *et al.* (2000) examined whether the cationicity of soluble dry strength agents had an effect on interfiber bonding. Their results showed that tensile index remained constant when the DS increased, and therefore concluded that there was no such effect on interfiber bonding. Pelton and Hong (2002) performed a similar study, impregnating newsprint with polyvinylamine with varying DS and came to the same conclusion, also

summarized in a review (Pelton 2004), that the charge of soluble dry strength agents affects the amount of polymer adsorbed onto the fiber, but has no effect on interfiber bonding.

This study investigated whether cationic groups chemically bound to lignocellulosic fiber fractions contribute to interfiber bonding, and thus paper strength, or whether the reported strength enhancement is instead related to side effects such as fiber flexibilization, not an exclusive property of cationic functionalities themselves. To eliminate or at least minimize the simultaneous effect of fiber flexibilization caused by the spacer effect, only the already highly flexible fines fraction (particles passing a screen with 100 μ m hole diameter) was cationized and added to the unmodified pulp or fractions thereof. Fines show a higher surface area than fibers. Assuming that only surfaces participate in bonding, any effect on strength should be even more pronounced for modified fines. To exclude the effect of improved retention because of the cationic charges, white water recirculation (Giner-Tovar *et al.* 2015) was used to guarantee comparable retention of unmodified fines to refined pulp (unmodified fines present), refined pulp with the fines washed out (no fines present), and unrefined fibers (also fines-free).

EXPERIMENTAL

Fractionation

Industrially bleached hardwood (refined and unrefined) and unrefined bleached softwood kraft pulp were fractionated. The fiber and fines fractions were produced with a lab-scale pressure screen at 1% consistency (Jagiello 2017). The pressure screen was equipped with a perforated plate (100 μ m hole diameter). The material passing through the plate was defined as the fines fraction. The pulp was recirculated until the remaining volumetric fines content (measured with an L&W Fibertester⁺, Lorentzen & Wettre, Stockholm, Sweden) was less than 0.5%. This fraction was defined as the fiber fraction. The highly diluted fines suspension was stored in large barrels to allow settling of the material. After 3 to 4 d, the supernatant was removed to obtain a fines suspension of approximately 1% consistency. The fiber fraction was filtrated to a consistency of 30%. The fractions obtained from the different pulps are summarized in Table 1.

Pulp	Fraction	Abbreviation
Unrefined bleached softwood kraft (spruce)	Fines	BSK _{fines} (cBSK _{fines})
Unrefined bleached hardwood kraft (eucalyptus)	Fibers	BHK _{fibers}
Refined bleached hardwood kraft	Fibers	rBHK _{fibers}
(eucalyptus)	Fines	rBHK _{fines}

Table 1. Pulps and Fractions Thereof, Abbreviations Used in this Study

Chemical Modification (Cationization)

Prior to cationization of the BSK_{fines}, acidic washing was conducted. The pH of the fines suspension, as achieved after pressure screening (approximately 1% consistency), was adjusted to a pH of 1 by adding HCl (4 N). After 1 h, the fines were filtered off and washed with deionized water until neutral. Then, 10 g (dry weight) of the fines and 22.2

mL of NaOH (4.5 N) were supplemented with deionized water to achieve a solids content of 1% fines and a concentration of 0.1 N NaOH in the reaction volume. After 30 min, 36 mL of 2,3 epoxypropyltrimethylammonium chloride (EPTMAC; Sigma-Aldrich, technical grade, 21.6% water, 90% purity of dry substance) were added to the stirred reaction volume. After 15 h at 40 °C, the reaction was stopped by adding enough HCl (4 N) to decrease the pH to approximately 1. The suspension was then filtered through a fine cloth in a Büchner funnel, and the solid material was washed with water until neutral. These cationized fines (cBSK_{fines}, Table 1) were again brought to 1% consistency. Further experimental details are published in Odabas (2016).

Degree of Substitution

The DS was determined by conductometric titration using a system by Metrohm (Herisau, Switzerland). An amount of the fines suspension corresponding to 100 mg of the dry sample was diluted to 50 mL prior to titration. During titration, 50 μ L of a 10 mM aqueous silver nitrate solution were added every 10 s (Odabas 2016). Triplicate measurements were performed. From the equivalence point (EP) (mL) and the dry mass of the titrated aliquot m_{dry} (mg), the DS was calculated, taking into consideration the concentration of the silver nitrate solution c_{AgNO3} (mmol mL⁻¹), the molar mass of one AGU (MM_{AGU} = 162 g mol⁻¹), and also the molar mass of the functional group (MM_{FG} = 152 g mol⁻¹), as described by Eq. 1,

$$DS = \frac{EP \cdot c_{AgNO3} \cdot MM_{AGU}}{m_{dry} - EP \cdot c_{AgNO3} \cdot MM_{FG}}$$
(1)

Sample Preparation

The fractions produced by pressure screening and cationization (Table 1) were blended as shown in Table 2 in order to obtain combinations with 5% unmodified BSK fines (Reference A-D), as well as combinations with 5% cationized BSK fines (A-D). The BSK fines were used because they showed a high water-holding capacity (more details: Mayr et al. 2017), and thus, they were considered to show a high conformability even before cationic modification. These fines were added to hardwood (BHK) pulp and fractions instead of the corresponding BSK fractions in order to minimize the effect of inferior sheet formation and thus, lower the variation in tensile measurement. The cationic fines were added to the refined pulp (refined fibers and fines), refined fibers (refined pulp without fines), and unrefined fibers (without fines), to see whether there were any differences depending on the addition of the cationized fines to unmodified fines (present in refined pulp), external fibrils still connected to the fibers (present in refined fibers with fines washed out), or plain fibers (unrefined fibers with fines washed out). For the combination with the refined pulp, the influence of mixing order was also evaluated by adding cationized fines (cBSK_{fines}) both to the refined pulp with unmodified fines present, and to the refined fibers (without fines) with subsequent addition of unmodified fines (rBHK_{fines}). In both cases, the composition of the final blend was the same, but any effects resulting from electrostatic interaction between the modified and unmodified fines dependent on the sequence of their addition should have become visible. To ensure a homogeneous distribution of fines, the suspension was mixed for 5 min with a kitchen blender with a flat rotating disk (1200 rpm, diameter 36 mm) directly after the addition of each fraction.

Table 2. Mass Proportion of Fractions in Blends and the Corresponding Mixing

 Order

		Composition (%)					
		rBHK _{fibers}	BHK _{fibers}	rBHK _{fines}	BSK _{fines}	cBSK _{fines}	Mixing Order
Refined Pulp	Ref. AB	86		9	5		rBHK _{fibers} +rBHK _{fines} +BSK _{fines}
	A	86		9		5	rBHK _{fibers} +rBHK _{fines} +cBSK _{fines}
	В	86		9		5	rBHK _{fibers} +cBSK _{fines} +rBHK _{fines}
Refined Fiber	Ref. C	95			5		rBHK _{fibers} +BSK _{fines}
	С	95				5	rBHK _{fibers} +cBSK _{fines}
Unrefined	Ref. D		95		5		BHK _{fibers} +BSK _{fines}
fiber	D		95			5	BHK _{fibers} +cBSK _{fines}

Sheet Preparation of Pulp and Paper for Testing

Drainability (ISO 5267-1 (1999), SR Tester, Gockel & CO) and zeta potential (Mütek SZP – 04, BTG Instruments) were measured directly on the blended samples. Handsheets of 60 g/m² were prepared (ISO 5269-2 (2004), Rapid-Köthen sheet former, FRANK-PTI) using white water recirculation. Following a method implemented by Giner-Tovar *et al.* (2015), the first five handsheets were discarded to ensure a stable and defined content of fines, independent of the given retention, being different for unmodified and cationic fines. For each fiber blend, eight handsheets were formed, and density (ISO 534 (2011)), tensile index, and elongation at break (ISO 1924-2 (2008), Tensile Tester, FRANK-PTI) were measured. Formation index was determined recording weight maps, captured in a Formex Betaformation Box (Science Imaging Scandinavia AB, Saltsjö-boo, Sweden), and subsequently scanned in a Fujifilm BAS 1800-II scanner (Fujifilm, Tokyo, Japan). An internal routine was applied on the weight maps to calculate a formation index, taking into consideration the intensity and size of the flocs.

RESULTS AND DISCUSSION

Whether cationic charges themselves led to increased paper strength was addressed by attempting to reduce side effects concerning the structural changes cationizing the fines fraction instead of the fibers. The pulp and sheet properties of the blends with cationized fines (A-D) were compared to their references (Ref. A-D), representing blends with the same amount of unmodified fines (Figs. 1, 3, and 4).

Differences in the consumption of the cationized fines by the unmodified fines, which may lead to changes in the pulp and paper properties, were considered by adding the cationized fines to refined pulp containing unmodified fines and external fibrils (black bars), to refined fibers containing only external fibrils still attached to the fibers (striped bars), and to unrefined fibers (dotted bars).

The chemical modification resulted in fines with a positive charge, *i.e.*, "cationized" fines with a DS of 0.034. Adding 5% of these fines to negatively charged pulp or fibers led, as expected, to a lower zeta potential (Fig. 1a). The effect on zeta potential was more

pronounced when adding cationic fines to the unrefined fibers (D), as fewer negative surface charges were available compared to the blends where unmodified fines (A, B) or refined fibers (C), having a higher surface area, were also present.

Electrostatic attraction between the positively charged fines and the negatively charged fibers or unmodified fines present in the pulp led to a reduction of drainage time (Fig. 1b) for all of the blends.



Fig. 1. Pulp properties: zeta potential (a) and drainage time (b); References (Ref. A-D) contained unmodified fines and (A-D) replacement of unmodified fines by cationic fines; black bars: fines were added to refined pulp (fibers + fines); striped bars: fines were added to refined fibers (fines removed); dotted bars: fines were added to unrefined fibers.

A combination of a certain flocculation of the fines material and an attachment thereof to the fiber surfaces was likely responsible for this decrease. This phenomenon is illustrated schematically in Fig. 2. Typically, both fines and fibers are negatively charged. Because of electrostatic repulsion, the fines would be quite uniformly distributed around the fibers (Fig. 2a). By replacing part of these unmodified fines with cationic-modified fines (Fig. 2b), the opposite charges are attracted, and therefore, cationic fines flocculate with unmodified fines and attach to the fibers (Fig. 2c). Less space is required and the fines no longer tend to plug the pores of the fiber mat, thereby reducing drainage time.



Fig. 2. Illustration of pulp suspension containing fibers and (a) unmodified fines; (b) replacement of unmodified fines by cationic fines and (c) its effect on flocculation, leading to lower dewatering resistance

The blend in which the cationic fines were added prior to the unmodified fines (trial point B) showed a faster drainage time than the blend in which the cationic fines were added after the unmodified fines (trial point A), although both blends were comprised of the same components (Table 2). In the case of trial point B, a higher proportion of cationic fines adhered to the fibers, as no other negative charges were available. The unmodified fines added afterwards then adhered to the fiber surface. In trial point A, the cationic fines could adhere to both the unmodified fines and fibers. Flocculated fines presumptively had

a higher potential to hinder dewatering, as they could fill up the pores of the formed fiber mat more easily than fines adhered to the fiber surface. The fact that these differences in flocculation were apparent, even after the application of high shear forces (1200 rpm) during sample preparation, indicates a strong adhesion and high shear resistance of the formed flocs.

Sheet density and formation index were not affected by the cationized fines addition in most of the blends (Fig. 3). An exception was sample C, where the decrease in density corresponded to an increase in formation index, indicating inferior sheet formation. This blend of fines-free, externally fibrillated fibers and cationic fines might have induced enhanced floc formation at positions of high external fibrillation because of the higher surface area available for electrostatic interaction with the cationized fines.



Fig. 3. Paper structural properties: apparent density (a) and formation index (b); for coding see Fig. 1.



Fig. 4. Paper mechanical properties: tensile index (a) and elongation (b); for coding see Fig. 1.

Overall, blends including cationic fines showed noticeably different behavior compared to the reference samples, influencing zeta potential, drainage time, and flocculation, as the result of the interaction of cationic groups with anionic-charged pulp components. However, no effects on tensile index or elongation at break were observed (Fig. 4), regardless of whether the cationic fines were added to pulp containing unmodified fines, refined fibers, or unrefined fibers. One might argue that soluble cationic dry strength agents, *e.g.*, cationic starch, improve strength properties. Pelton (2004) investigated this effect and found that it was not cationicity itself that was responsible for the strength agent. He concluded that the electrically charged groups were mainly responsible for retention, but

not for strength development. In the case of soluble dry strength agents, the sheets containing them are typically compared to sheets without dry strength agents. In these experiments, the reference sheets contained the same amount of fines as the sheets with the modified fines. Considering the different setup, effects similar to the addition of soluble dry strength agents were also visible in a higher strength when an additional 9% fines (trial setup A-B compared to setup C) was added.

Similar to the cationic fines, an experiment adding cationic MFC (Gao *et al.* 2016) also gained no improvement in tensile strength. These observations do not support the findings for cationic pulps or fibers, where obviously improved strength properties were reported.

It seems reasonable to argue that the improvement in paper strength properties observed by other authors as the result of cationization of pulp or fibers was related to the spacer effect (Gruber 2002), increasing fiber flexibility and thereby conformability. Similar effects were also observed in a study by Zhao et al. (2016) in which the carboxyl group content of mechanical pulp fibers was increased. This treatment resulted in higher fiber flexibility and thus, improved strength properties. The scheme of a fiber cross-section and the outermost fiber wall of both unmodified (Fig. 5a) and cationized fibers (Fig. 5b) illustrate the proposed effect of cationic charges acting as a spacer in the fiber wall, and thereby improving paper strength. Negative charges are already present in the fiber wall and on the surface of unthreaded fibers (Fig. 5a). In the direct proximity of these charges, hydrogen bonding is hindered. The light areas of the illustration of the outermost fiber wall indicate regions that are loosely bound because of charged groups. Upon increasing the content of charged groups by cationization, the fiber wall may become more delaminated and thus, the fiber more flexible. This spacing would also take place on the fiber surface, increasing the loosely bound region of the outermost fiber wall and enhancing conformability.



Fig. 5. Illustration of fiber cross-section and the possible effect of charges on the topology of the outermost fiber wall; tightly bound areas (dark red), loosely bound areas (light red); unmodified fibers negatively charged (a), and cationized fibers (b), proposed effect of cationic charges acting as a spacer in the fiber wall.

In addition to fiber flexibilization and the resulting improved sheet consolidation, cationic groups also promote retention. The associated strength improvement comparing sheets from cationic pulp (fibers + fines) with sheets from untreated pulp might also be related to improved fines retention, especially for mechanical pulps showing a high proportion of fines (Montplaisir *et al.* 2006).

Although cationic groups do not improve strength properties intrinsically, strength improvement is possible with cationizing fibers due to flexibilization of the fiber without mechanical treatment. Taking into consideration the other positive effects of cationization, such as improved runnability and cleanliness of the system, improved filler and fines retention, improved drainage, reduced beating-resistance because of the spacer effect, and even improved internal sizing adding only small amounts of cationic fibers (Krause *et al.* 1981; Gruber 2002), cationization of fibers might nevertheless be an interesting option for upgrading fibers and stock compositions.

CONCLUSIONS

- 1. Sheets containing cationic fines showed differences in zeta potential, flocculation behavior, and drainage time, compared to sheets containing the same amount of unmodified fines, revealing the activity of cationic groups.
- 2. Nevertheless, cationic fines did not have a significant effect on paper strength improvement, which is in accordance to the findings concerning cationic polyelectrolytes.
- 3. Therefore, it might not be the cationic groups *per se*, but rather the increased fiber flexibility that is responsible for the strength-enhancing effect seen in several studies on cationic pulp and pulp fibers.

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