

## THE INFLUENCE OF CATIONIZED BIRCH XYLAN ON WET AND DRY STRENGTH OF FINE PAPER

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Cationized birch xylan was prepared and its use as a papermaking chemical was evaluated. The focus was on studying the effects of cationized birch xylan on the wet and dry strength of fine paper. The results of the laboratory experiments show that the addition of 3 percent of cationized birch xylan to birch kraft pulp improved the initial wet strength of the web by 30 percent compared to base stock at a solids content of 55%. Furthermore, the tensile stiffness of the wet web increased by approximately a third and the dry tensile strength improved by 26%, while the dry elastic modulus was not changed. The improvements in the strength properties were clear when compared to the base stock, but not as high as achieved with conventionally used cationized starch. The difference between the xylan and starch is most likely due to the shorter polymer chain length of the cationized xylan.

**Keywords:** *Cationized birch xylan; Initial wet strength; Strength; Tensile stiffness; Runnability, Fine paper*

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## INTRODUCTION

Good web runnability in papermaking and printing processes is dependent primarily on the machine-directional strength properties of the paper web. In the paper machine, the most critical point is the zone between the press section and dryer, where the web passes through open draws. In this zone, depending on the raw material base used, the solids content of the web ranges from 35 to 55 percent and, according to the widely held view, the web strength is derived mainly from the adhesion of water menisci at the fibre crossings and mechanical friction forces between the stock components (Page 1993).

The straining of the wet web generates plastic deformation even at low tensions and, consequently, strongly influences paper properties such as porosity. High tensile stiffness of the wet web is therefore desirable. With respect to dry paper, an extensive analysis of press room runs has shown that high MD tensile strength together with high structural uniformity correlate with low breaking frequency in press rooms (Uesaka et al. 2001; Deng et al. 2004).

The strength of both wet and dry paper can be increased by the addition of chemical additives to the stock. The additive most commonly used is cationized starch, due primarily to its positive effect on the dry strength of paper, where it is thought to increase both the bonded area and the strength of the inter-fibre bonds (Howard and

Jowsey 1989; Stratton 1989; Laleg et al. 1991). As a positive side effect, cationized starch also improves retention during web formation and may also contribute to better paper machine runnability by increasing the rewetting rate of the web. Other chemicals used for improving initial wet strength include cationic aldehyde starch, carboxymethyl cellulose (CMC), and glyoxylated cationic polyacrylamide (G-PAM) used in tissue production (Salminen 2010).

The use of wood-derived xylan as a papermaking chemical has not been widely studied. Depending on the species, wood typically consists of 25 to 35 percent hemicellulose. The main types of hemicellulose in wood are glucomannan and xylan, and their contents depend on the wood species: softwoods have higher glucomannan contents and lower xylan contents, whereas in hardwoods, the ratio is reversed. Xylan is composed primarily of xylose units and exists as arabinoglucuronoxylan in softwoods and as glucuronoxylan in hardwoods, the latter accounting for 15 to 30 percent of hardwood dry mass.

The degree of polymerization of wood hemicelluloses is relatively low, usually in the range of 50 to 200. The molecular weight of xylan is considerably lower than that of starch, which, depending on the ratio of amylose to amylopectin and their respective chain lengths, has a  $M_w$  of hundreds of thousands to millions.

The influence of xylan addition on pulp beatability as well as paper strength properties and optical properties has been studied by Ramírez et al. (2008) and Köhnke and Gateholm (2007). The former group experimented with arabinoxylans from corn cob and oat spelt to examine their sorption to bleached and unbleached softwood kraft pulp. The results indicated that the addition of xylan prior to the refining of pulp is the most effective method of xylan application to promote fiber development (fibrillation, wet-conformability, etc.) and strength properties. The tensile and burst indices are improved by xylan addition over the whole range of beating degrees. In contrast, tear strength is increased significantly for unbeaten pulps, but declines after beating, compared to the reference. Xylan sorption rates are increased with prolonged beating. Ramírez et al. concluded that the strength increase alone will not usually justify the application of xylans as an alternative to common papermaking additives.

The focus of Köhnke and Gateholm's research was on the influence of glucuronoxylan on hornification and the location of adsorbed xylan. They concluded that xylan adsorption occurs predominantly on the fibril surfaces within the fibre after diffusion into the porous fibre wall. Treatments resulted in increased tensile strength and beatability, whereas the tensile strength at a given °SR, density, and light scattering coefficient were more or less unaffected.

The objective of the present study was to prepare and evaluate cationized birch xylan as a process and functional chemical in papermaking. Xylan was extracted from bleached birch pulp under alkaline conditions and reacted with glycidyltrimethylammonium chloride (GTAC). The focus was on studying the effects of cationized xylan on the wet and dry strength of paper made with fine paper furnish. In order to verify the fixative effects, the target for the xylan DS of was set above 1. The performance of cationized xylan in regard to the paper properties was assessed in relation to a chemical-free base stock as well as conventionally used cationized starch.

## EXPERIMENTAL

### Materials

The glycidyltrimethylammonium chloride (GTAC) was supplied by BASF (Raisacat, 73% solution in ethanol), and was used without further purification. Other reagents were purchased from Sigma-Aldrich and used as received.

Alcofix 169 (solution, D.S.C. 47.5%), used as reference during fixative studies is a medium molecular mass, linear poly(diallyldimethylammoniumchloride), distributed under the brand name Alcofix 169 from Ciba Specialty Chemicals Co. Dosages during tests were 0.5 mg/g, 1.0 mg/g, and 2.0 mg/g.

Fennopol (K3400R, powder), used as a retention aid in sheet forming, was acquired from Kemira. The stock solution concentration was 0.3%. The retention aid dosage was 0.02 percent. Cationic wet-end starch (Raisamyl 135, DS 0.035) from Raisio Chemicals (nowadays Chemigate) was used at dosages of 1 and 3 percent (wrt total fibre content). Precipitated calcium carbonate filler (SYNCARB S-PCC-IM 35%) from Omya was used at a target content of 25%.

### Methods

#### *Alkaline extraction of birch xylan*

Alkaline extractions of xylan from bleached birch pulp (ECF, Kaskinen mill) were carried out at VTT's Rajamäki pilot plant (1 M NaOH, consistency 5.5 percent per hour at 25 °C). The slurry was first separated using an Alfa-Laval NX309 Decanter centrifuge. The cellulose was then washed with water and treated with biocides. Next, the alkali was removed from the xylan-containing water phase by ultrafiltration (Alfa-Laval 6.3-3 PN, Spiral Gross-Flow, surface area 15 m<sup>2</sup>, cut-off 10,000 MW). A 6 percent solution was prepared and spray-dried using a Niro P6.3 spray dryer. According to the results, approximately 53% of the existing xylan (12.5–23.6 percent xylan from polysaccharide) was extracted from the birch pulp. The purity of the xylan was very high (96.7 to 97.6 percent xylan of total sugars).

#### *Preparation of cationized xylan*

100 g of xylan (0.76 mol), 315 mL of GTAC (1.5 mol), 140 mL of H<sub>2</sub>O (7.5 mol), and 4.6 g of NaOH (0.12 mol) were mixed thoroughly in a reaction flask. The mixture was then stirred in a water bath at a constant temperature of 45 °C overnight. The mixture was then washed with ethanol and H<sub>2</sub>O, and filtered. The mixture was finally ultrafiltrated using a membrane with a cut-off of 3,000, and samples for NMR analysis were dried in vacuum. The reaction yields a white powder with a yield of 73% of the original xylan recovered. See Fig. 3 for numbering. <sup>13</sup>C NMR (125 MHz, D<sub>2</sub>O) δ: 57.1 (C9), 65.8 (C5), 68.0 (C6), 71.1 (C7), 75.7 (C8), 76.6 (C4), 79.3(C2/C3), 84.5 (C2'), 104.7 (C1) ppm. The total nitrogen content was 54.800 mg/kg (Kjehldahl titration).

#### *Characterization using NMR*

The solution state <sup>13</sup>C NMR spectra of the cationic xylan were recorded using a Bruker 500 MHz Avance III NMR spectrometer with a 5.0 mm BBO probe operating at 125.8 MHz. All NMR experiments were carried out at room temperature. 10000 scans

and a delay time (D1) of 5.0s were used for the experiments. The sample for NMR analysis was prepared by first freeze-drying the cationic xylan (80 mg), and then solubilising it in D<sub>2</sub>O (1.0 mL) at room temperature.

#### *Experiments for testing the fixative effects of cationized xylan*

The fixative experiments were carried out using TMP pulp that was hot disintegrated at a consistency of 1.5%. The fibre fractions were measured with an L&W fibre tester, with the fines fraction defined as particles shorter than 0.2 mm. The disintegrated pulp was further diluted to a consistency of 1 percent and mixed at a temperature of 60°C for 3 hours in order to release anionic trash from the fibres in dissolved or dispersed form into the process water. Next, the studied fixatives were added, and the pulp was mixed for 10 min. The cationized birch xylan was dosed at levels of 0.5, 1.0 and 2.0 mg/g. Finally, the pulp was centrifuged with 1700 rpm for 15 min, and the filtrate was taken for analysis. The principal measurements for characterizing the xylan derivatives were turbidity (adapting ISO 7027), charge density (according to VTT internal instruction of PRO52048), and conductivity as well as pH (according to VTT internal instruction of PRO52101). A commercial product, Alcofix 169, was used as the reference. The pulp pH before the fixative tests was 5, and the pH was not adjusted.

#### *Measurements for predicting the web runnability*

The experiments for testing the papermaking effects of cationized xylan consisted of making laboratory sheets (adapting SCAN-CM 26:99) of fine paper stock and testing the resulting sheet properties. The base stock was unrefined birch kraft with PCC filler at a 25% target content (filler content was determined adapting SCAN-C 6:62). The pulp suspension pH was adjusted to approximately 8, using sulphuric acid. The conductivity was adjusted to 1000 µS/cm, using sodium chloride. In addition, Fennopol K3400R was used as a retention aid at a 0.02 percent dosage. The addition of cationized xylan was 3% (30 mg/g). An additional experiment was carried out with a high dosage of 10% cationized xylan and an elevated dosage of retention aid (0.2%). Cationized starch was used for comparison and was also combined with cationized xylan at a dosage of 1%. The tested mixes are presented in Table I.

**Table I.** Experimental Design for Testing the Effects of Cationized Xylan on Papermaking Properties \*

	Birch kraft pulp (% of fibre content)	PCC (% addition wrt fibre content)	Retention aid (% of total dry weight)	Cationized xylan (% of total dry weight)	Cationized wet-end starch (% of total dry weight)
Reference	100	25	0.02	-	-
Starch	100	25	0.02	-	1.0
Cationized xylan	100	25	0.02	3.0	-
Cationized xylan +starch	100	25	0.02	3.0	1.0
Cationized xylan at high dosage	100	25	0.20	10.0	-

\* The values are percentages of dry solids; the filler is calculated in relation to the fibre base (birch), while the other additions are relative to the sum of fibre and filler.

The target basis weight of the sheets was 60 g/m<sup>2</sup> (grammage was measured according to SCAN-P 6:75 and thickness in line with SCAN-P 7:75) and the sheets were prepared with a circulating water sheet mould. The sheet mould drainage time was measured in order to detect potential differences in inter-fibre dewatering. Optical properties of studied dry paper samples were measured adapting SCAN-P 3:93 and SCAN-P 8:93.

Dry paper strength was determined with a Lloyd tensile tester (adapting SCAN-P 38:80 and SCAN-P 67:93). The tensile strength and relaxation properties of the wet paper were measured with an Impact fast tensile strength test rig (Fig. 1). In this experiment, the paper sample length was 100 mm and the strain velocity 1 m/s (1,000%/s). The width of the wet samples was 20 mm. The relaxation properties for wet paper were measured at 2% constant strain.

During the impact measurements, the samples were attached between two jaws. The lower jaw was then moved to a set position to generate strain. The upper jaw of the impact rig is equipped with a load sensor, and the amount of strain was measured with a laser sensor. The measurement was done at two dryness levels, and 10 to 14 parallel samples were measured. At each dryness level, 4 to 6 samples were taken for determination of dry solids content, and the dryness of the paper samples was determined using a Mettler Toledo HR73 infra-red dryer. The validity of each test result from the Impact device was tested using Dixon Massey criteria (SCAN-G 2:63).



**Fig. 1.** Impact test rig for measuring the strength properties of wet papers. The output consists of tensile strength, strain at break, dynamic tensile stiffness, and residual tension

## RESULTS AND DISCUSSION

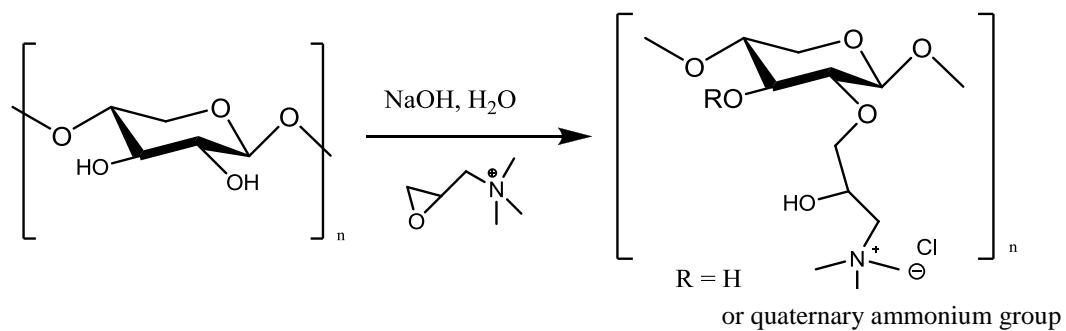
### Isolation and Cationization of Xylan

The molar masses of the extracted xylan were determined in three different eluent systems, producing some variation depending on the used method (Table 2).

**Table 2.** Molar Masses of the Extracted Birch Xylan in Three Different Eluent Systems

	$M_n$	$M_w$	PD
DMSO:water (90:10)	13610	19910	1.5
DMSO	14360	23300	1.6
0.1 M NaOH	13730	19850	1.4

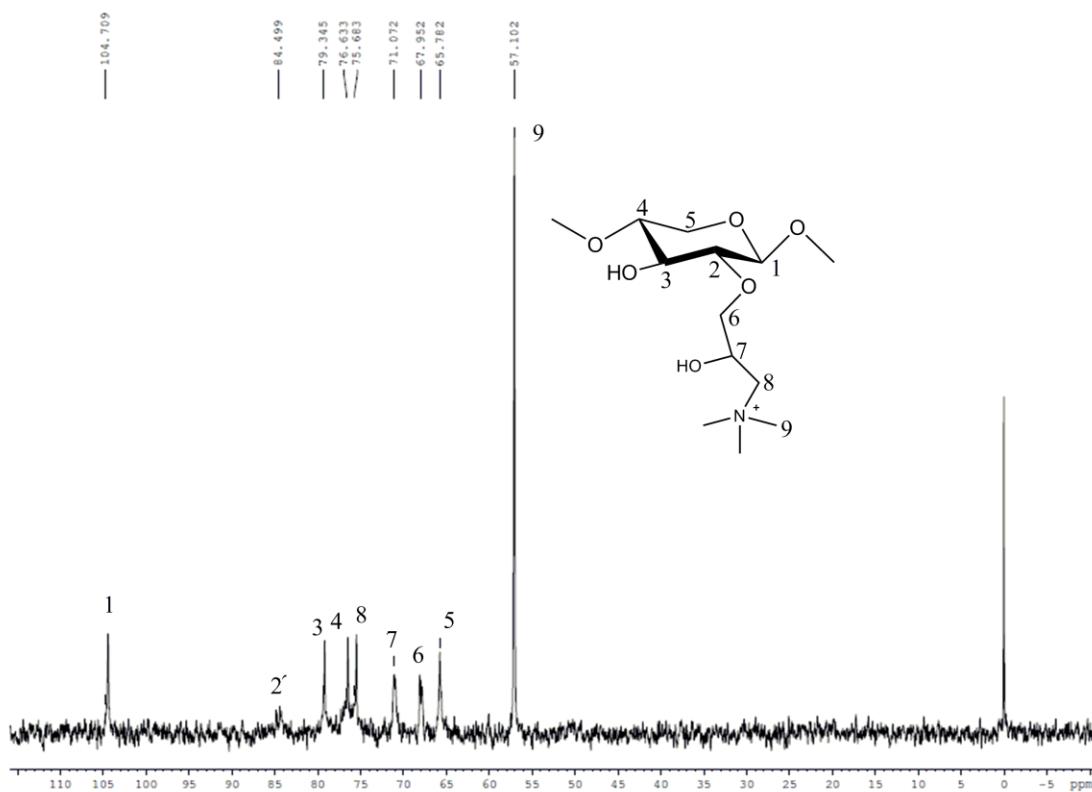
Cationic xylan was prepared using a modified reaction route described in the literature (Bendoraitiene et al. 2006) for the cationization of starch. The synthesis is presented in Fig. 2. The reaction mechanism relies on strict control of the amount of water present in the reaction mixture. The nitrogen content was analyzed using Kjehldahl titration, and calculation of the degree of substitution, according to literature (Bigand et al. 2011, Ebringerova et al. 1994), yielded a degree of substitution of 1.27.

**Fig. 2.** Cationization of xylan

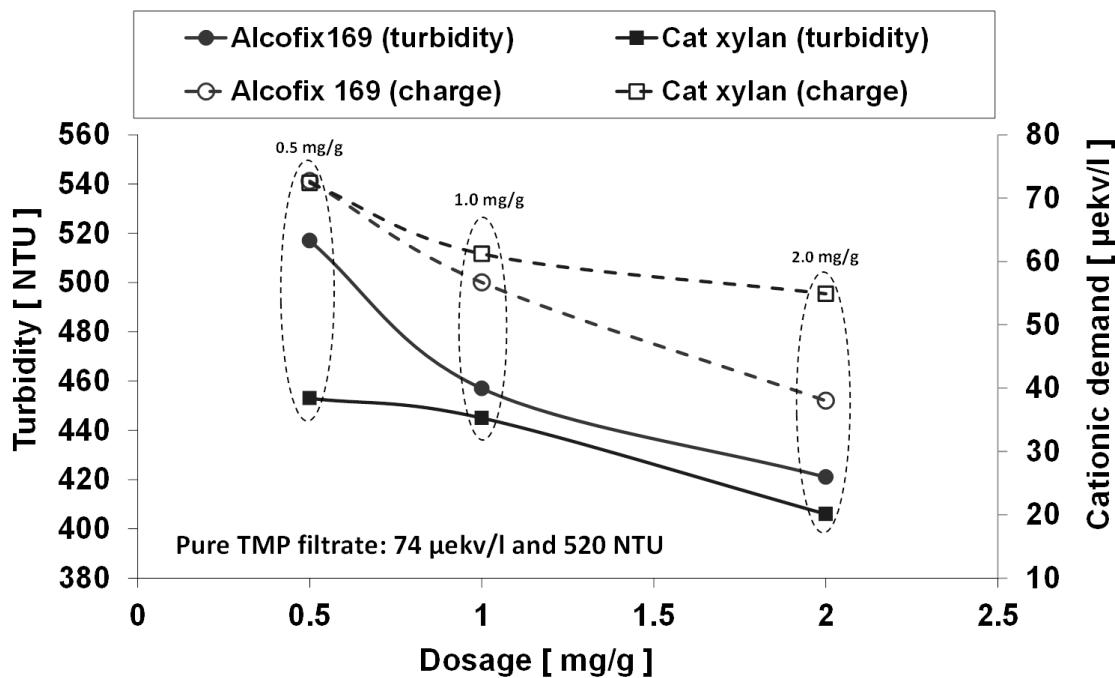
Cationic xylan was characterized using  $^{13}\text{C}$ -NMR spectroscopy. The spectrum (Fig. 3) is in complete accordance with similar spectra presented in the literature, and the signals were similar and thus assigned accordingly (Ebringerova et al. 1994; Ren et al. 2008; Yan et al. 2009; Schwikal et al. 2006). Specific peak assignments are presented in detail in Fig. 3. The signal of the C2 carbon in the xylan backbone is shifted downfield due to the addition of the cationic substituent. A similar behaviour was described by Ebringerova et al. (1994). The degree of substitution was determined by integrating the relative peak area of the cationic substituent carbon C9 and comparing it with the peak area of the xylan backbone C1. A similar method has been used for example by Hettrich et al. (2006), Ren et al. (2008), and Liu et al. (2005). The received degree of substitution was 1.27, which is in accordance with the result obtained using Kjehldahl titration.

### Papermaking Effects of Cationized Xylan

The action of cationized xylan in the pulp-water suspension was first examined with mechanical pulp to see if it would absorb into the fibrous components of the stock. For the experiment, the cationized xylan was added to TMP with a fines fraction content of 27.6%. In these conditions, the cationic component should neutralize part of the anionic charge of the stock and, consequently, flocculation of fines should occur.



**Fig. 3.** <sup>13</sup>C NMR spectrum of cationic xylan

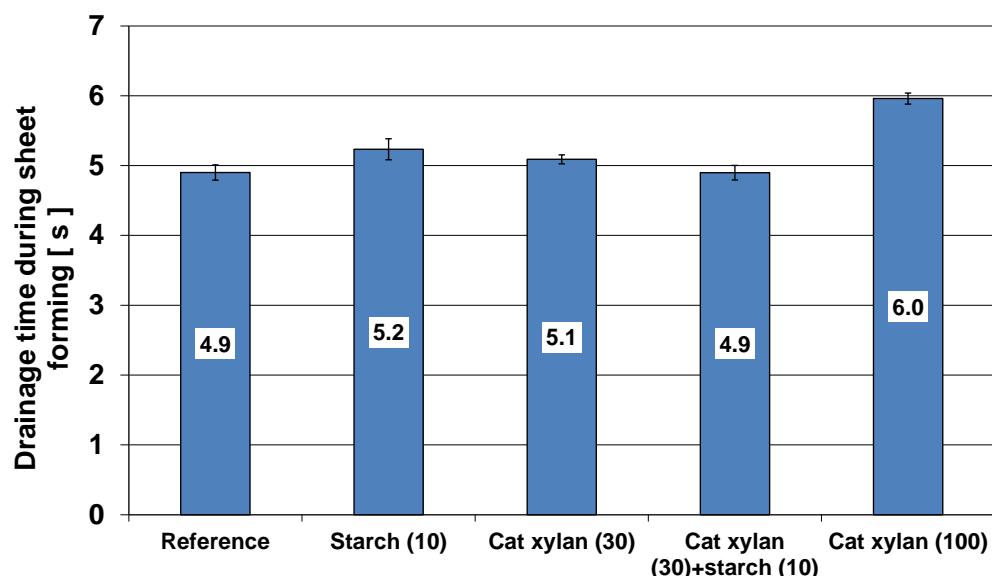


**Fig. 4.** Turbidity and charge level of TMP filtrates after addition of commercial fixative and cationized xylan

As shown in Fig. 4, the turbidity and charge level, measured as cationic demand, both decreased, showing that partial neutralization of the anionic charge of the pulp components has occurred. The effect is similar to the reference fixative (Alcofix 169), although the effect on charge level is less pronounced at the highest dosage level.

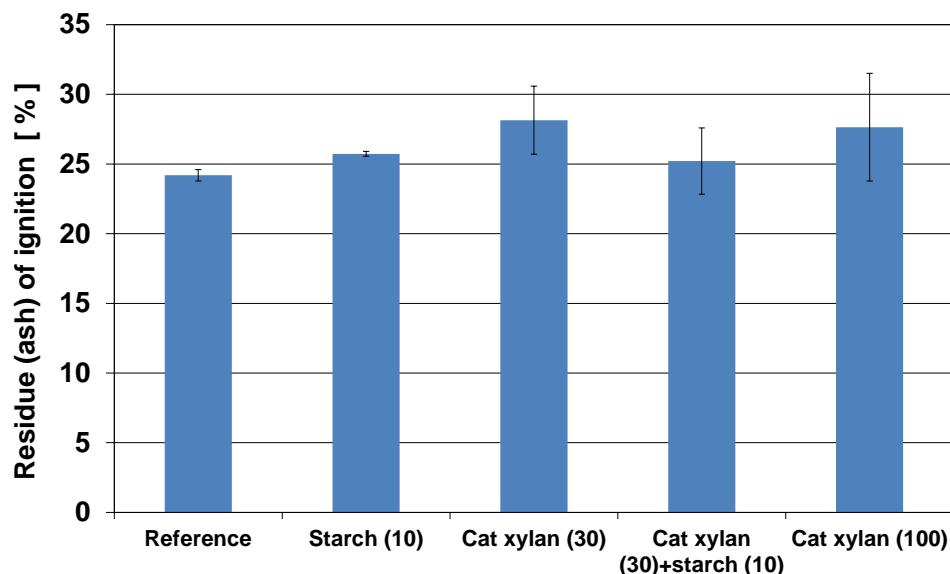
In the next phase of the experiments, fine paper stock was used as described in the Materials and Methods sections. The drainage times monitored during handsheet forming are presented in Fig. 5. The values were similar except for the high dosage of cationized xylan. The drainage times indicate that there are no differences in inter-fibre dewatering between the stocks, suggesting that the addition of xylan does not increase the net hydrodynamic surface area of the stock, but there is a neutralization of anionic charges and possibly adsorption of polymer into the cell wall. The latter explanation is in accordance with the conclusions of other studies (Köhnke and Gatenholm 2007), stating that xylan adsorption occurs primarily on the fibril surfaces within the fibre after diffusion into the porous fibre wall.

In practice, the result means that the solids content of the web after the forming section should be approximately the same with constant settings for the dewatering elements. A high dosage level of cationized xylan is an exception, producing a clearly slower dewatering rate, but as the addition level is unrealistically high, the result is irrelevant in this regard. The slower dewatering could result from increased filtration resistance produced by a more complex pore structure of the web as an excess of highly cationic polymer imparts a positive charge by its adsorption onto all of the surfaces in the system. Repulsion between like-charged surfaces causes fines to be dispersed from surfaces and causes fibrillation to extend outwards from the fibre surfaces, thus tending to impede flow through the fibre mat. The un-attached fines also tend to impede flow by moving relative to the adjacent fibres and becoming mechanically trapped at restricted points in the spaces between fibres in the mat.

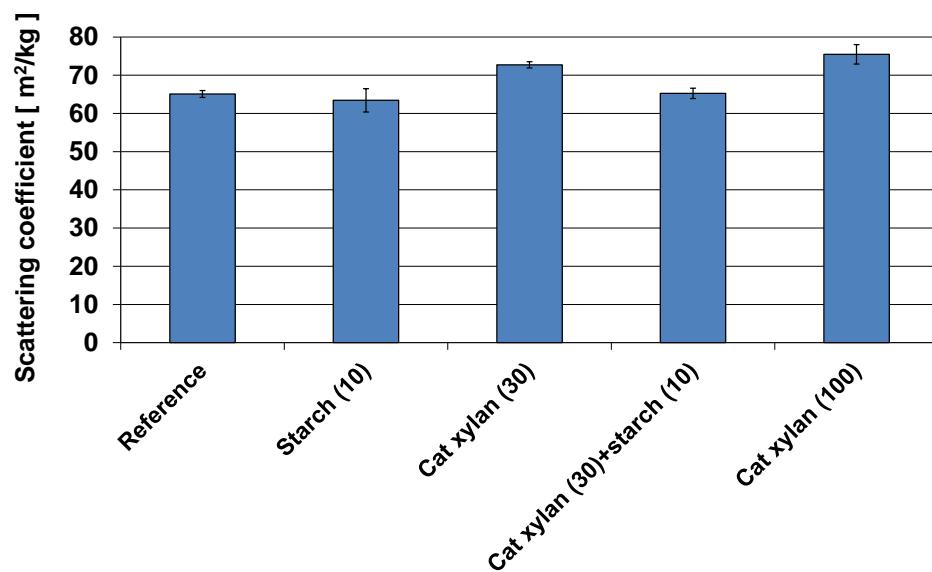


**Fig. 5.** Drainage times of the studied samples (added chemical amounts (kg/t) shown in parentheses)

For measuring the actual filler content of the sheets, the residue (ash) of ignition was determined (Fig. 6). As the graph shows, the 25% target was exceeded by 2.5 to 3 percentage points in the samples with cationized xylan addition. As the filler has an impact on the strength of the paper – the higher the filler content the lower the strength - this must be taken into account in the interpretation of the results. In this case the difference is relatively small, and it should not produce significant differences in the web structure in regard to the strength properties, thereby allowing the comparison of the results.



**Fig. 6.** The ash content of the fine paper sheets was slightly above target (25%) in papers with cationic xylan addition



**Fig. 7.** The light scattering coefficient values measured in dry papers reflect the small differences in filler content

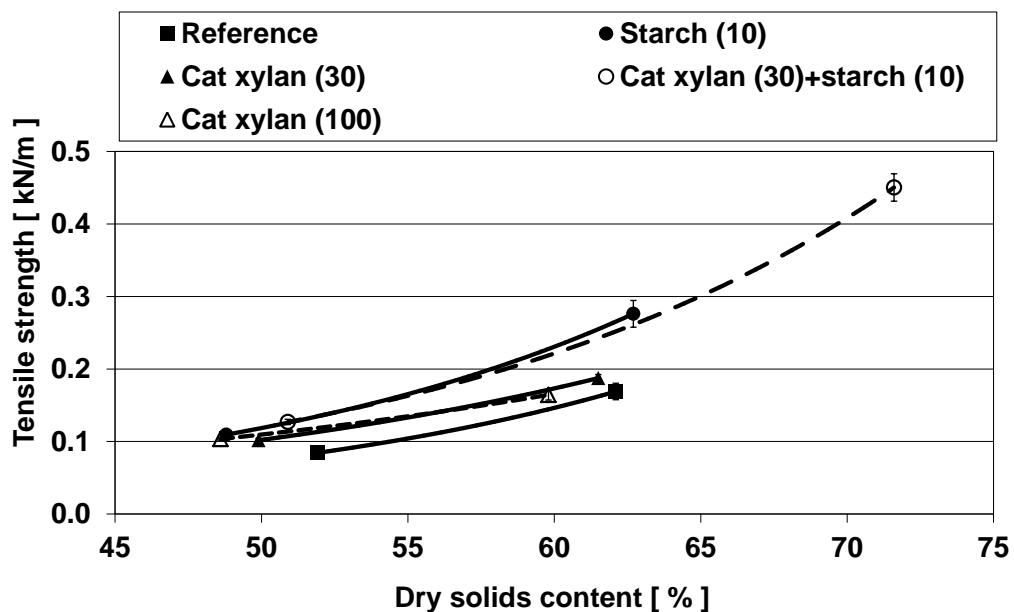
The higher filler content of these sheets is also the reason for their comparatively high light scattering coefficients (Fig. 7). A probable reason for the higher filler content could be a filler dosing error, although it is also possible that the cationized xylan has a positive impact on filler retention.

Initial wet tensile strength increased with the addition of both cationized xylan and starch, as seen in Fig. 8. At a solids content level of 55%, the base stock had a tensile strength of approximately 100 N/m. A 3% addition of cationized xylan increased this value to 130 N/m, while a 1 percent addition of cationized starch yielded a strength of approximately 165 N/m. There was no further increase in strength when cationized xylan was added together with starch, nor with an excessive xylan dosage of 10%. It should be noted that the samples to which both xylan and starch had been added had undergone excessive wet pressing, resulting in a clearly higher solids content of the web. However, this does not affect the strength comparisons of the samples at 55% solids content.

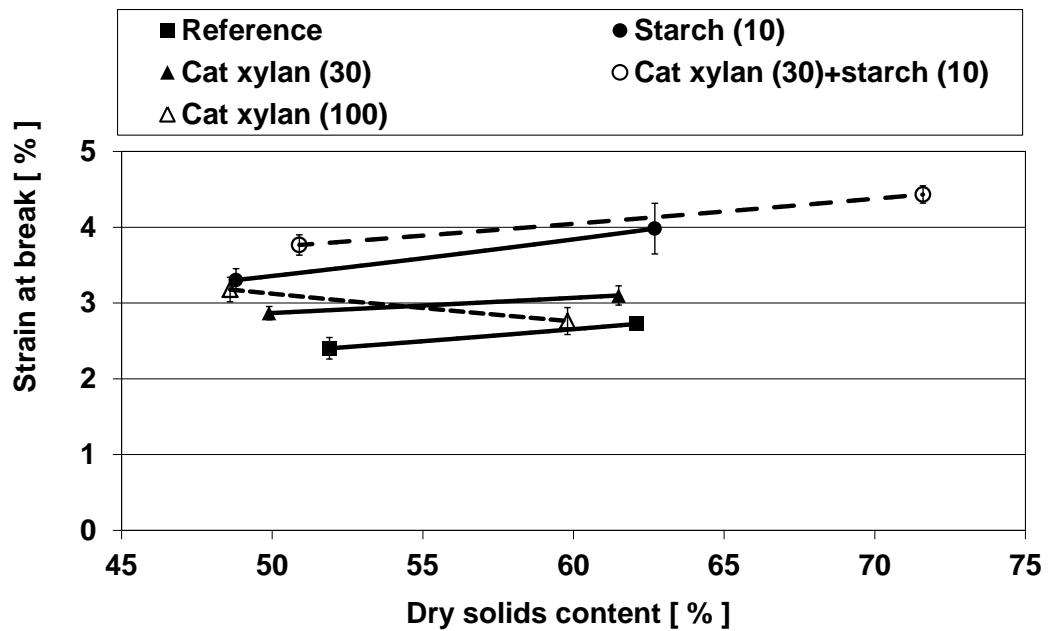
Increased wet tensile strength allows the web to withstand higher tension peaks produced by the process and, consequently, should bring a reduction in the frequency of web breaks at the press section and at the start of the dryer section. The probable reason for the wet strength increase is the enlarged contact area between fibres as the adsorbed polymers make the fibre surface more irregular in shape, resulting in increased tangential adhesion between fibres. In addition, the polymer addition may increase the viscosity of the liquid phase, again contributing to higher adhesion forces. The differences in the performance of starch and xylan can be mostly explained by the differences in the polymer chain lengths: shorter xylan produces less new contacts between particles than longer-chained starch. Depending on the eluent system used, the measured molecular weight ( $M_w$ ) of the isolated xylan is in the range of 19850 to 23300, while unmodified starch has an  $M_w$  of over a million. Although the cationization lowers the  $M_w$  of starch somewhat, it is still considerably higher than in birch xylan or its cationized form.

There were also differences in the breaking strains of the wet samples, as shown in Fig. 9. At 55% solids content, starch addition increased the breaking strain from 2.5 to slightly below 4%, while cationized xylan raised the breaking strain to ca. 3%. As the strain measurement of wet samples often produces high variation in this range of solids content, the significance of the strain differences should be interpreted with caution.

The tensile stiffness of the wet papers was of a different order compared to the ranking for tensile strength (Fig. 10): at 55% solids content, the reference sample had a tensile stiffness of ca. 10.5 kN/m, whereas the cationized xylan increased the value to 14 kN/m. Contrary to tensile strength, starch addition produced lower tensile stiffness than xylan, at ca. 12 kN/m. The residual tension showed smaller differences between the reference and the papers with chemical additions, as seen in Fig. 10. At 55% solids, the values were almost within the margins of error. A higher wet elastic modulus enables stable passage of the wet web over the open draws with smaller speed differences between rolls and cylinders. Significant to dryer section drying, xylan and starch addition decreased the solids content of the web compared to the base stock when examined at the same wet pressing pressures. The difference with xylan was ca. 2 percentage points, while starch decreases the solids content by approximately 3.5 percentage points. This should be taken into account when assessing the overall runnability effects, as the energy consumption in the dryer section is dependent on the solids content of the incoming web.



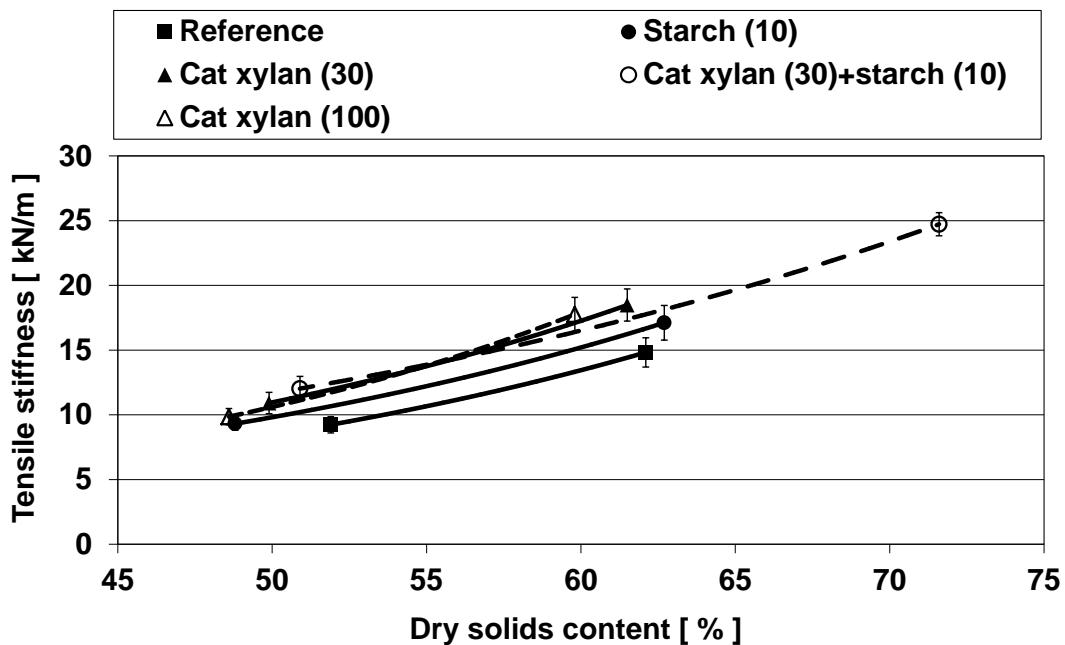
**Fig. 8.** Wet paper tensile strength values of the measured samples. Measurement was performed at two solids content levels, adjusted by wet pressing pressure. An exponential function has been fitted to each data pair.



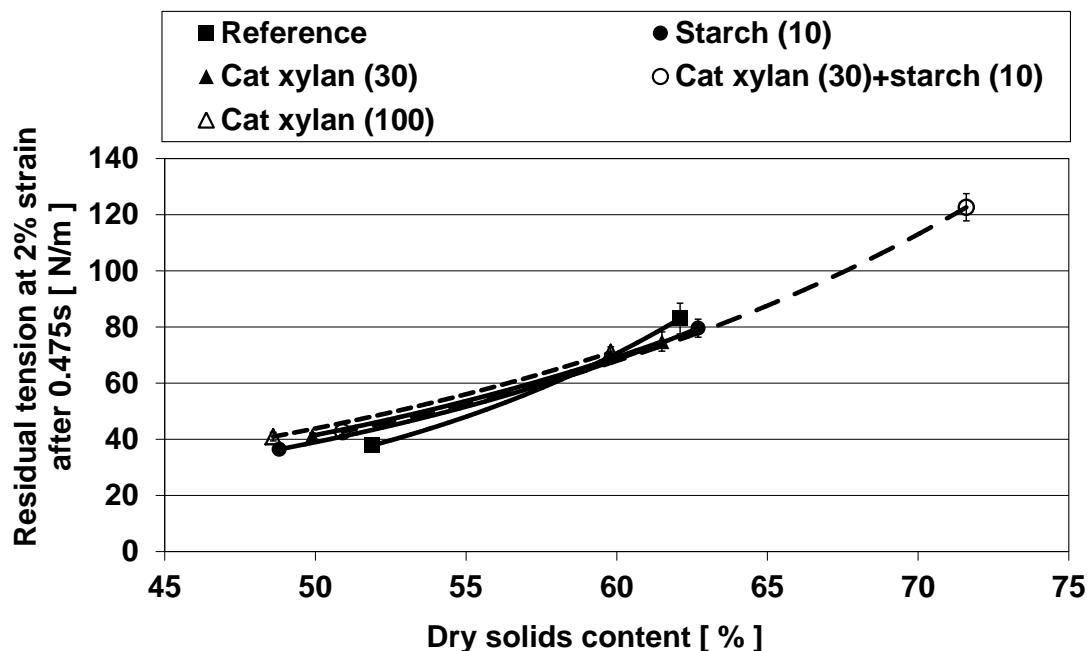
**Fig. 9.** Strain at break values with linear fits for wet papers

The changes in dry paper strength are also clear, as presented in Fig. 12: starch addition increased the tensile index by almost 85% and cationized xylan by 26%. The combination of starch and xylan produced the same strength as starch alone. Increasing the xylan dosage from 3 to 10% had no influence on the tensile index. Both starch and

cationized xylan addition increased dry paper strain at break (Fig. 13). As regards modulus of elasticity (Fig. 14), only starch addition had an influence on the paper, while cationized xylan had the same value as the reference.

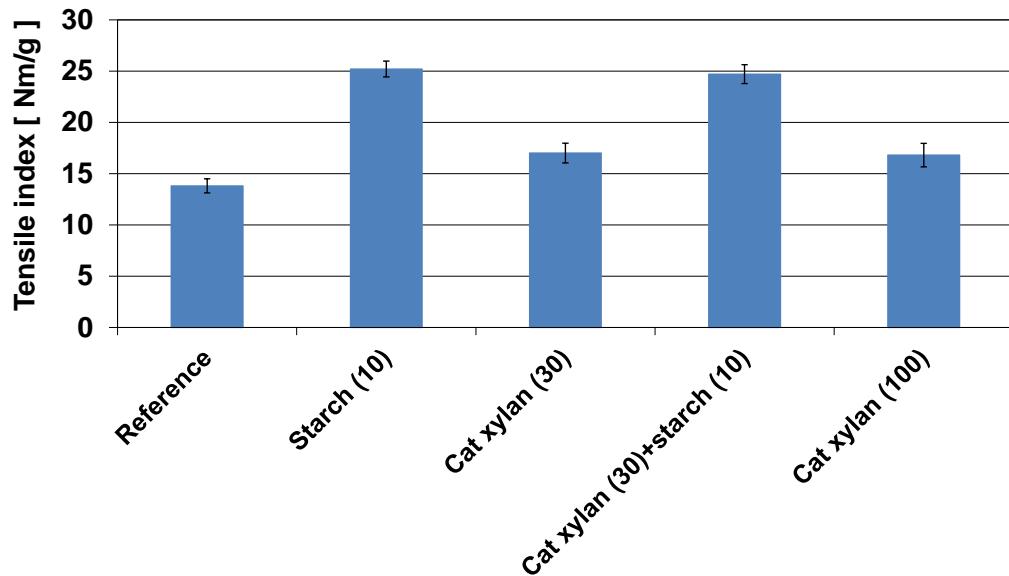


**Fig. 10.** Wet paper tensile stiffness values of studied samples with exponential fits

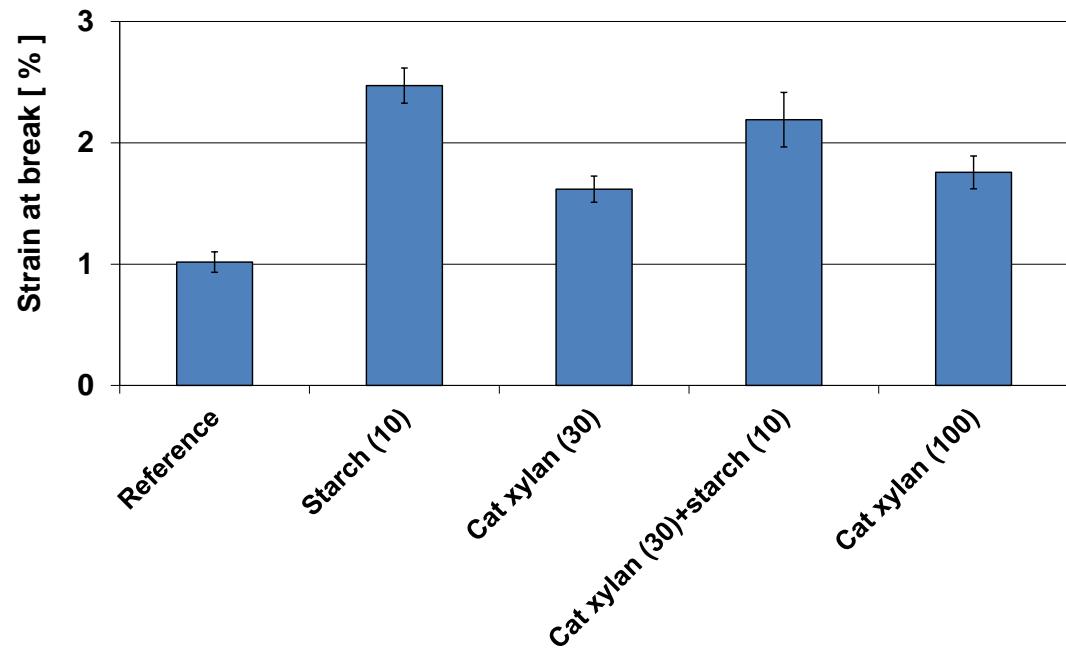


**Fig. 11.** Wet paper residual tension values with exponential fits

With the addition of cationized xylan the tensile strength increase was evident, although the elastic modulus did not change compared to the reference paper. Taking into account the filler content, which is slightly higher in the cationized xylan containing sheets compared to other test points, we may speculate that its impact on strength would be more pronounced at an equal filler content (see Fig. 6).



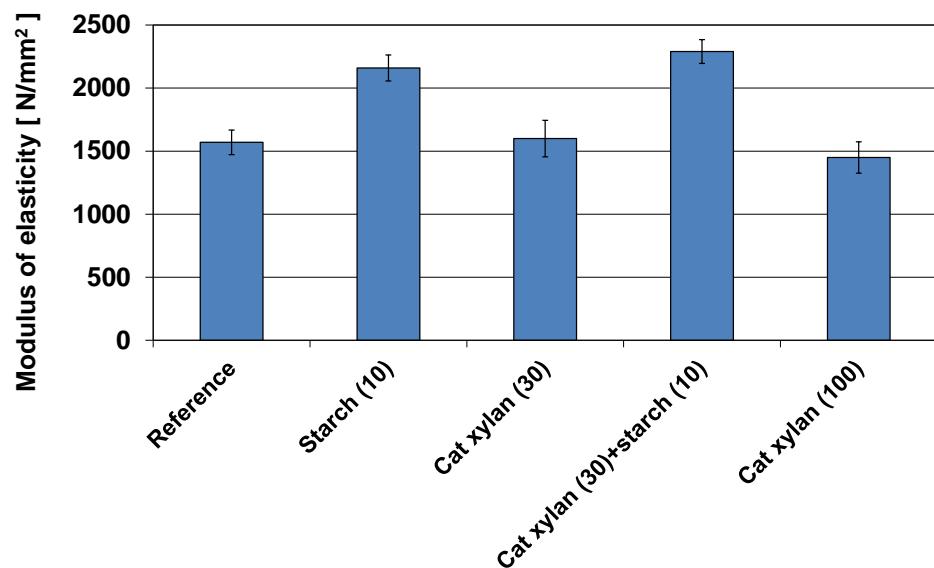
**Fig. 12.** Dry paper tensile index values of the studied samples



**Fig. 13.** Dry paper strain at break values of the studied samples

Starch addition clearly increased the strength, and the increase was significant in terms of both tensile strength and elastic modulus. As far as starch is concerned, the mechanism creating higher strength is thought to be enlarged bonded area together with stronger bonds between fibres. Again, the differences between the cationized xylan and better performing starch are probably due to the greater reach of starch polymers, which create a larger contact area between the fibres and lead to the generation of more numerous inter-fibre bonds.

As presented above, the results of the tested cationized birch xylan show statistically significant improvements in important paper properties. However, it is difficult to justify cationized xylan addition based on these improvements alone. Considering the cost of xylan extraction and employment compared to the use of conventional chemicals, e.g. cationized starch, the economy of using xylan as a replacement wet-end fixative and paper chemical would be questionable, and requires further clarification. However, the experiments do indicate that hemicellulose-based papermaking chemicals do have potential, and with further modification – as well as potential changes in the availability and cost of currently used chemicals – these derivatives could be developed as competitive paper chemicals. External factors, such as food price inflation, could, for example, lead to a situation where the use of edible vegetable based additives would be economically less viable for industrial purposes.



**Fig. 14.** Dry paper elastic modulus values of the studied samples

## CONCLUSIONS

- With respect to the important paper properties, the most important positive effect of the addition of cationized xylan, as compared to the reference case where no strength increasing chemicals were used, is a clear increase in wet strength properties. In addition, the tensile strength of dry paper increases with the addition of cationized

birch xylan. However, the overall performance of cationized birch xylan in terms of both wet and dry paper properties is poorer than conventionally used cationized starch, and, considering the related costs, it is difficult to justify xylan addition based on the measured quality improvements alone.

2. The differences between cationized xylan and the better-performing starch are due to the greater reach of the longer starch polymers, which create a larger contact area between fibres, leading to superior inter-fibre bonding.
3. The decrease in TMP filtrate turbidity and cationic demand clearly show that cationized xylan interacts with the fibre component and contributes to web properties. Although with birch kraft pulp the addition of cationized birch xylan does not increase the net hydrodynamic surface area of the stock, neutralization of anionic charges does occur, possibly along with partial adsorption of the polymer into the cell wall.

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