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## Surface Sizing with Starch Solutions at Solids Contents up to 30%

### Summary

In this work, we have studied surface sizing of uncoated fine paper with a metered size press (MSP) unit at considerably higher starch solids content than conventionally used (< 10%). This work had a specific aim to increase the solids content of the starch solution from 21% to 30% and keep the applied dry starch amount as close as possible to 1.5 g/m<sup>2</sup>/side. The machine speed used in the study was 1200 m/min. Two different modified starch types (e.g. anionic and cationized) were selected with different viscosity levels of 20 mPas and 100 mPas (60 °C, 10%).

With the low starch penetration, a high surface strength of the paper was reached. Additionally, the air permeability and oil absorption are considerably reduced compared to the basepaper values, since the starch remains on the surface. An interesting observation was the fact that using the low viscosity starch, the internal strength was high compared to the base sheet even at the highest solids content levels. The development of internal strength even at very high starch solids values opens new possibilities in optimizing the final paper properties when high solids surface sizing is used.

An increased solids content of the starch solution at a constant dry starch amount reduces the required evaporation energy and drying capacity. The starch viscosity needs to remain unchanged at an increased solids content or compensated with a smaller rod diameter. Less wetting of the paper web at higher starch solids also reduces the number of web breaks at the MSP unit, which then improves the total net efficiency of the paper machine. Additionally, surface sizing with high solids starch solutions makes it possible to use the same process conditions (e.g. evaporation capacity and roll covers) for both sizing and coating operations.

### 1. Introduction

The main role of the surface size is to promote surface properties, e.g. strengthen the surface and to bind particles such as fibers and fillers to the surface. Additionally, the starch is expected to add internal strength to the sheet through a liquid penetration in the z-direction<sup>1</sup>.

By using high solids starch solutions, a reduced evaporation requirement and an increased paper quality can be achieved. It is then necessary to maintain the runnability and the film forming capability when applying thinner starch films on the roll surfaces. Starches used for surface sizing are degraded and often additionally chemically modified by introduction of functional groups<sup>2</sup>. There are many modification methods, such as oxidation, thermal modification and ionization. In the MSP metering and transfer stages, certain starch properties are required, such as its ability to remain stable even at high solids contents<sup>3</sup>. De-

grading the starch molecules gives certain runnability properties, but if a starch molecule is broken down too much, the binder properties will decrease with lower ability to enhance the desired paper properties. Also, its ability to work as a carrier for optical brighteners may decrease<sup>4</sup>. Here, instead of just degrading the starch molecules, a combination of certain functional properties can both give starch both desired stability level and provide ability to promote desired paper properties even at very high solids contents.

In respect of desired starch properties at a high solids content, a combination of controllable rheological properties of the starch solution in combination with a required binding potential is the main target of this work. The metering process is critical since the leveling and overall quality of the film directly influences the paper properties. Here, the rheological properties of the starch will be crucial in terms of its stability<sup>5,6</sup>.

A number of studies has been carried out on surface sizing – even at high solids contents – with the MSP technique e.g.<sup>7,8,9,10,11</sup>. Lipponen et al<sup>12</sup> reported MSP pilot trial results using increased (8–18%) starch solids content at a constant dry starch amount, which resulted in increasing surface strength and bending stiffness. These observations, as well as the decrease in oil absorption, air permeability and internal strength were explained through the decreased starch penetration.

However, the surface sizing at solids content level from 20 to 30% is not well covered in the literature. This study is then performed as an attempt to further inquire the surface sizing possibilities using very high starch solution solids content levels.

### 2. Experimental

#### 2.1 Materials

##### 2.1.1 Starch

Two modified potato-based starches (Raisio Chemicals Inc., Raisio, Finland) were modified with a specific focus on solution stability even at very high solids contents.

A native potato starch was initially modified through oxidation with hypochlorite to a carboxylic groups content of about 1% (DS<sub>-COOH</sub> = 0.031). The viscosity of this starch A (Raisamyl 01020) was 20 mPas (Brookfield 100 rpm, 60 °C) at a solution solids content of 10%. The cationization was done through introduction of quarternary ammonium groups followed by oxidation. The degree of substitution (DS<sub>-NH<sub>4</sub></sub>) was 0.018. The viscosity of the cationized starch B (Raisamyl 24520) was 100 mPas (60 °C) at a solution solids content of 10%. The starch properties are summarized in *Table I*.

Table I: Properties of the starch solutions.

Property	Solids content (%)	Starch A	Starch B
Viscosity, Brookfield 100 rpm at 60 °C (mPas)	21	65	207
	24	80	338
	27	125	–
	30	158	–
Average molecular weight, M <sub>w</sub> (g/mol)	27,000	~ 50,000 <sup>1)</sup>	
Degree of polymerization, DP	150	300	
Degree of substitution, DS	0.031 <sup>2)</sup>	0.018 <sup>3)</sup>	

<sup>1)</sup> Not measurable with the used columns used in the Size Exclusion Chromatography (SEC)

<sup>2)</sup> Degree of substitution of carboxyl groups (DS<sub>-COOH</sub>)

<sup>3)</sup> Degree of substitution of quarternary ammonium groups (DS<sub>-NH<sub>4</sub></sub>)

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**2.1.2 Base paper**

A woodfree base paper without surface sizing at a basis weight of 71 g/m<sup>2</sup> was used for the study. The properties of the base paper are presented in *Table II*.

*Table II: Properties of the base paper*

Properties		Values
Grammage	g/m <sup>2</sup>	71.0
Ash content	%	10.0
Internal sizing	%	0.8
Density	kg/m <sup>3</sup>	738
Air permeability, Bendtsen	ml/min	709
Oil absorption, Cobb-Unger (10 sec)	g/m <sup>2</sup>	34.3
Surface strength, IGT (MD)	m/s	–
Internal strength, Scott Bond	J/m <sup>2</sup>	305
Bending stiffness, MD	mNm	0.63
Bending stiffness, CD	mNm	–

**2.2 Methods**

**2.2.1 Process conditions**

The trials were run as a C2S MSP application (OptiSizer, Metso Paper Inc.) at a speed of 1200 m/min and a linear load of 25 kN/m. The roll cover material was polyurethane with a hardness of 36 P&J. The roll diameters were 950 mm. The film metering was performed with 10 mm smooth rods. The wet film on the roll surface was measured<sup>13</sup> to determine the rod diameter requirements to reach starch amount level of 1.5 g/m<sup>2</sup>/side.

A single nip soft calender was used at a machine speed of 1200 m/min with a steel roll temperature of 60 °C and a linear load of 45 kN/m. The hardness of the polymer roll was 90 ShD with a roll diameter of 420 mm. Topside of the paper was run against the steel roll.

**2.2.2 Measurements**

The paper properties were measured according to the standards reported in *Table III*.

*Table III: Measurement methods and standards*

Methods	Standard
Grammage	SCAN-P 6:75
Ash content	SCAN P 5:63
Density	SCAN-P 7:96
Air permeability, Bendtsen	SCAN-P 60:87
Oil absorption, Cobb-Unger (10 sec)	SCAN-P 37:77
Surface strength, IGT	SCAN-P 63:90
Internal strength, Scott Bond	TAPPI T833 om-96
Bending stiffness	SCAN-P 64:90

The rheological characterizations of the starch solutions were performed at temperatures of 20, 30, 40 and 50 °C for each solids concentration (e.g. 21, 24, 27 and 30%). The flow in the low shear rate region (1–1500 s<sup>-1</sup>) was studied with a rheometer based on a Couette viscometric flow, where the inner cylinder is a stator and the outer cylinder rotates (Bohlin VOR rheometer, Bohlin Inc., Lund, Sweden). A yield stress,  $\tau_B$ , describing the stress needed to obtain flow was calculated for the starch solutions according to the Bingham plastic flow model. An intermediate shear region (10<sup>3</sup>–10<sup>5</sup> s<sup>-1</sup>) was studied for the starch solutions with a rheometer of a Searle flow geometry, where the inner cylinder rotates and the outer cylinder is a stator (Hercules, HiShear rheometer, Kaltec Scientific Inc., Detroit, MI, USA). An air-pressure region corresponding to a high shear region of 10<sup>5</sup> s<sup>-1</sup> to 10<sup>7</sup> s<sup>-1</sup> was used in the air-pressure driven capillary viscometer (ECV, Gradek Oy, Grankulla, Finland), where the controlled shear stress measurements are done according to a

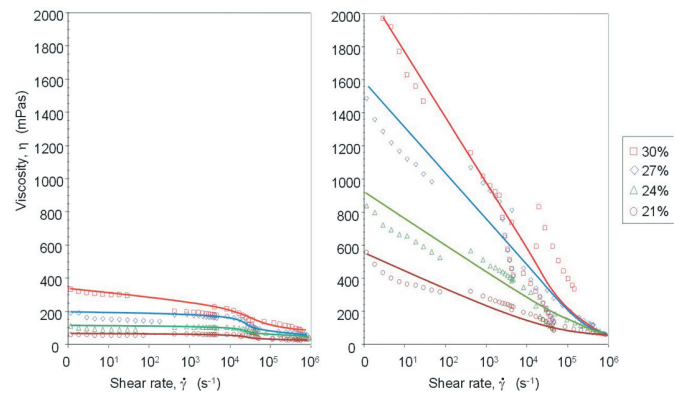
Hagen-Poiseuille viscometric flow. Stainless steel capillaries were used with a length of 100 mm and a diameter of 0.55 mm. The data were corrected for entrance effects according to the Hagenbach-Couette and end-wall effects according to the Rabinowitz-Weissenberg equation<sup>14</sup>.

Total starch amount applied was measured from the paper samples using a lithium chloride analysis<sup>15</sup>, where a weight portion of the sample is burned in a muffle furnace. The residue is dissolved with acids and the individual element concentration is then determined with ICP-AES (e.g. Inductively Coupled Plasma Atomic Emission Spectroscopy).

**3. Results**

**3.1 Effect of solids content on the flow properties of the starch solutions**

The flow properties of the two starch solutions were determined in a shear rate region from 10<sup>0</sup> to 10<sup>6</sup> s<sup>-1</sup> with 21% and 30% solids at 40 °C (*Figure 1*). The reason for presenting the rheometer results in temperature of 40 °C was the fact that it was difficult to maintain higher temperatures in the capillary during the measurement. It



*Figure 1: Effect of the solids content ( $\phi_s = 21, 24, 27$  and 30%) of the starch solutions on the viscosity ( $\eta$ ) at shear rates ( $\dot{\gamma}$ ) from 100 s<sup>-1</sup> to 10<sup>6</sup> s<sup>-1</sup> (40 °C) with a low viscosity starch A (left) and a high viscosity starch B (right).*

was found that the main viscosity difference was given by the initial modification differences, where a higher molecular weight for the cationized starch B gave a more pronounced shear-thinning flow. It is obvious that the low viscosity starch A did not show similar shear-thinning effect. In the shear rate region of 10<sup>6</sup> s<sup>-1</sup>, the viscosity differences between the two starch types are small at all solids content levels. One could suggest, that – as film forming mechanism using smooth rod can be seen as a hydrodynamic phenomena with the high shear viscosity as a major contributing factor<sup>16</sup> – similar film amounts should be reached with both starches during comparable conditions, despite of the large initial viscosity difference. The flow stability during the measurement – even at the highest shear rates – appears to have a stable shear thinning behavior throughout the whole shear rate region. Also one can observe, that there is a drop of viscosity level seen in the shear rate region of 10<sup>4</sup> s<sup>-1</sup>–10<sup>5</sup> s<sup>-1</sup> with the low viscosity starch A. This may rather be related to differences in used measurement methods in the shear rates of 10<sup>3</sup> and 10<sup>5</sup> s<sup>-1</sup> than an actual change in rheological properties of the starch solution in the shear rate region mentioned. The different high shear viscosity behavior at a shear rate of 500,000 s<sup>-1</sup> is further visualized in *Figure 2*.

It was expected that the starch molecules would create a weak network, which could develop an initial yield stress prior to breaking. At low solids content level (8–18%)<sup>12</sup>, the yield stress has been

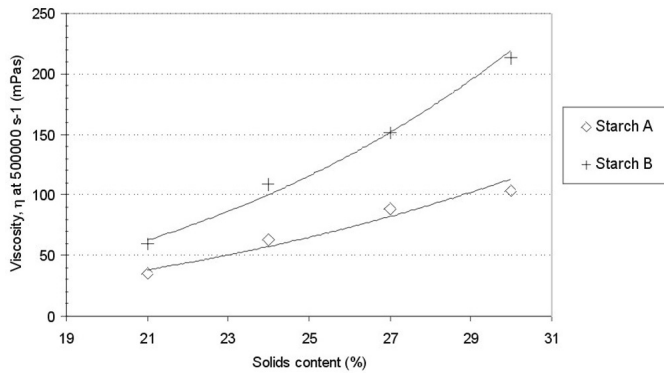


Figure 2: Effect of the solids content of the starch solutions ( $\phi_c = 21, 24, 27$  and  $30\%$ ) on the viscosity ( $\eta$ ) at a shear rate ( $\dot{\gamma}$ ) of  $500,000\text{ s}^{-1}$  at a temperature ( $\theta$ ) of  $40\text{ }^\circ\text{C}$  with a low viscosity starch A and a high viscosity starch B.

found to be low for both starch solutions, which was seen to correlate well with the excellent film forming, coverage and weak starch molecule network (e.g. low yield stress values). However, with starch solids contents of 21–30%, higher yield stress values were measured especially with the high viscosity starch B (Figure 3). During the trial, it was observed that an increased web release force was required when using the high viscosity starch B at solids content of 24%. Here, the release distance of the web on the bottom roll increased up to 200 mm. With starch solution solids contents of 27 and 30% of the high viscosity starch B the web was not able to be released from the bottom roll. The Starch A however was able to be released up to 30% solids content. When comparing the web release behavior of the different solids contents and starch types, one can suggest a correlation with a required level of yield stress level of the starch. Here, a yield stress level of below 400 mPa would provide a sufficient polymer mobility for stable web release in the MSP roll nip (Figure 3).

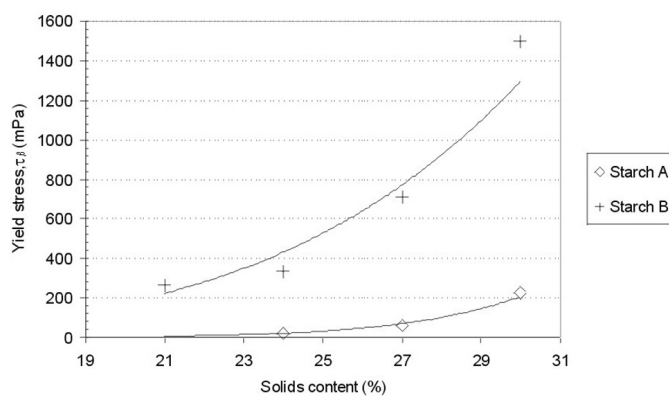
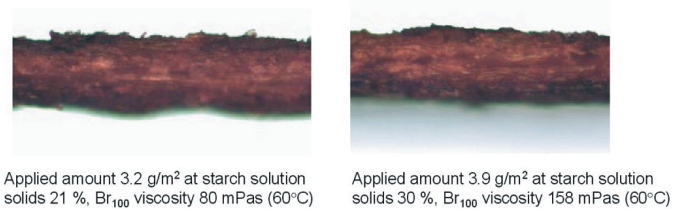


Figure 3: Effect of the solids contents ( $\phi_c = 21, 24, 27$  and  $30\%$ ) of the starch solutions on the yield stress ( $\tau_e$ ) at a temperature ( $\theta$ ) of  $40\text{ }^\circ\text{C}$  with a low viscosity starch A and a high viscosity starch B.

### 3.2 Effect of starch solution solids content on the starch penetration

By increasing the solids content of the starch solution from 8% to a level of 21–30%, there is less starch penetrating and then more starch remaining on the paper surface. With this level, the penetration of starch is almost negligible at all the studied starch solids content levels. Figure 4 presents the cross sections of paper samples with starch amounts of 3.2 and 3.9 g/m<sup>2</sup> at solids contents of 21 and 30%. It can also be observed however, the starch seems to have locally penetrated to the larger pores of the sheet also at the highest starch solids as well.



Applied amount 3.2 g/m<sup>2</sup> at starch solution solids 21%, Br<sub>100</sub> viscosity 80 mPas (60°C)

Applied amount 3.9 g/m<sup>2</sup> at starch solution solids 30%, Br<sub>100</sub> viscosity 158 mPas (60°C)

Figure 4: Cross-sections of surface sized paper with 3.2 and 3.9 g/m<sup>2</sup> of total starch amount at 21 and 30% solids content a low viscosity starch. The iodine stained starch can be seen as darker areas in the pictures.

### 3.3 Effect of starch solution solids on the paper properties

There is no major change in the surface strength as the solids content is changed from 21% to 30%. As the starch is already concentrated to the paper surface at a solids content of 18%<sup>12</sup>, no significant increase can be seen (Figure 5).

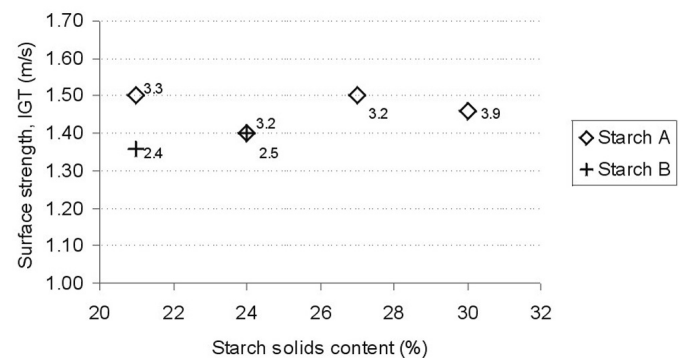


Figure 5: Effect of solids content on the surface strength (IGT) with two starch amounts A and one of B. The starch amount values are presented next to points.

The porosity of the paper decreases with 60–80% compared to the base sheet, when high solids starch solution was applied to the paper (Figure 6). With both starches, the air permeability was reduced from 700 ml/min to 275–100 ml/min at the highest solids contents. With the high viscosity starch solution, a more complete sealing of the sheet was obtained due to the reduced starch penetration. Another factor contributing to the lowered air permeability with the starch B may be partly explained by a more complete film forming capability with the longer molecular chains.

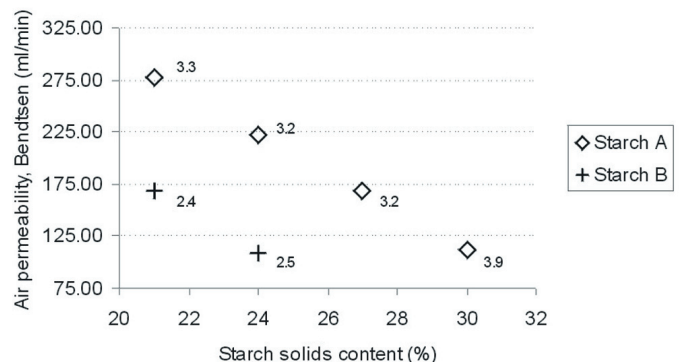


Figure 6: Effect of solids content on the air permeability (Bendtsen) with two starch amounts A and one of B. The starch amount values are presented next to points.

At an increased starch solids content, the oil absorption is reduced (Figure 7). A reduced openness of the paper surface with a higher starch concentration is naturally the explanation to this result.



Especially, the high viscosity starch B gave a clear oil absorption reduction from 34 g/m<sup>2</sup> for the base sheet to below 7 g/m<sup>2</sup> as the starch solids content was increased up to 30%. With the lower viscosity starch A, the oil absorption was also decreased to 7 g/m<sup>2</sup>.

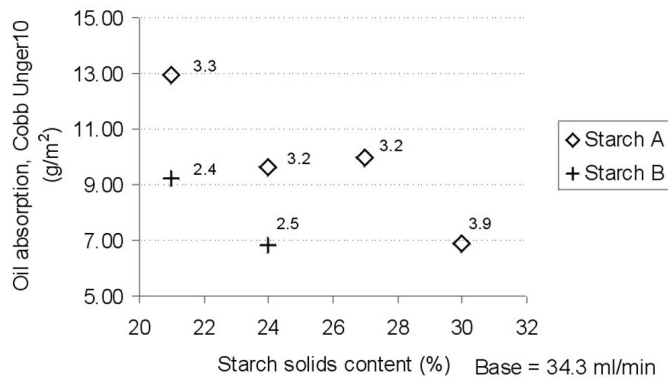


Figure 7: Effect of solids content on the oil absorption (Cobb-Unger,  $CU_{10}$ ) with two starch amounts A and one of B. The starch amount values are presented next to points.

As expected, the internal strength (e.g. Scott Bond) decreases as a result of a decreased starch penetration at increased starch solids (Figure 8). Starch B with a higher viscosity remains more on the surface compared to starch A and therefore the internal strength is more reduced. However, it was a bit surprising that the high solids surface sizing had a positive effect on the internal strength on the base sheet – contrary to earlier studies (e.g. Lipponen *et al*<sup>12</sup>).

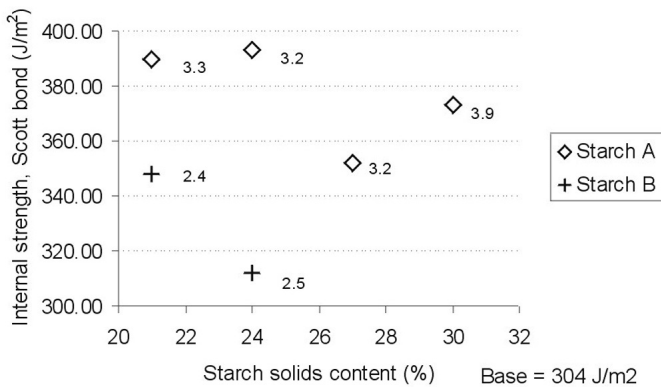


Figure 8: Effect of solids content on the internal strength (Scott bond) with two starch amounts A and one of B. The starch amount values are presented next to points.

One could explain it by pores size/penetration behavior with the lower viscosity starch even at the highest solids content level. The development of internal strength reported here at increased solids content is a bit contradictory to the previous<sup>12</sup>, where an almost negligible increase of base sheet internal strength was reported with 18% starch solids for a 82 g/m<sup>2</sup> base sheet. This result may also be explained through the lower basis weight of the base sheet, which may promote the development of the internal strength even at low starch penetration. Lowering the base sheet basis weight below 80 g/m<sup>2</sup> is known to increase Scott bond values due to decreased probability of potential rupture planes during Scott bond measurement<sup>18</sup>. Also, the increased starch amount used at 30% solids content most probably has an effect to the increased Scott bond values. This observation agrees well with the findings of Küstermann also<sup>8</sup>, who reported increased Scott bond values at increased starch solids content levels, when he increased the starch amount at the same time.

The influence of solids content on bending stiffness shows no significant change as starch solids is increased towards 30% (Figure 9). In the literature (e.g.<sup>17</sup>), increased bending stiffness is reported with increased starch solids, explained with starch being more concentrated on the surface of the sheet. Bending stiffness behavior with different starch solids contents in machine direction was similar, with the values of bending stiffness in the level of 0.45 mNm.

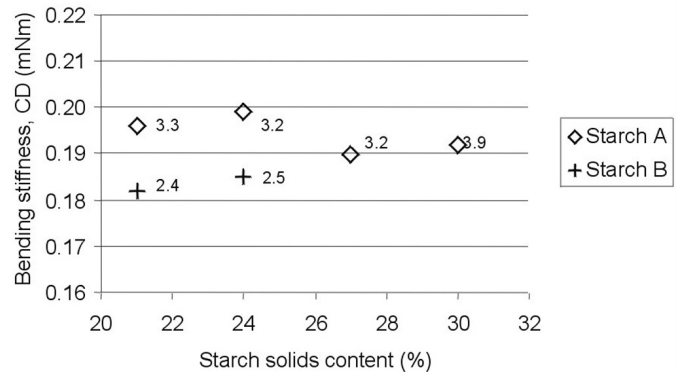


Figure 9: Effect of solids content on the cross direction bending stiffness with two starch amounts A and one of B. The starch amount values are presented next to points.

A suggested explanation to the decreased bending stiffness can be found in Figure 10, where bulk values of all measured trial points are plotted against starch amount at different solid contents. According to these measurements, bulk of the sheet is decreased as a function of the applied starch amount, almost regardless of the solids content. Here, the starch amount is increa-

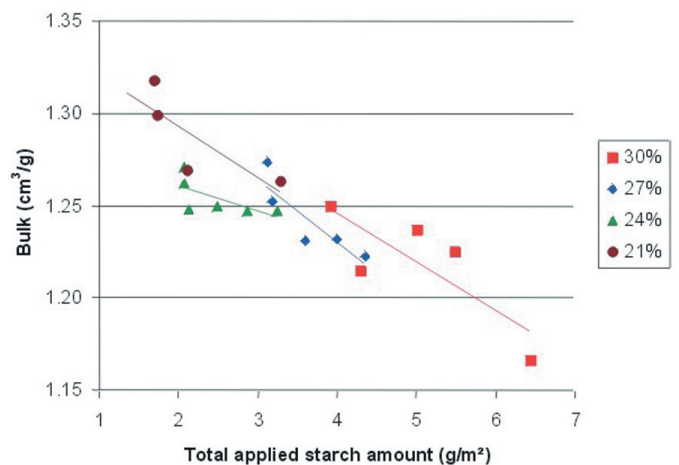


Figure 10: Effect of solids content on the bulk as a function of the total applied dry starch amount with the low viscosity starch A.

sed especially when a 30% starch solution is applied. When considering the third power interdependency of paper thickness (~ bulk) and bending stiffness<sup>18</sup>, an explanation can be suggested for the observed bending stiffness reduction at the highest solids contents. The CD stiffness variations are, however, dominated by the MD fiber orientation.

#### 4. Discussion

As reported, a solids content of up to 30% is possible to apply without remarkably increasing the dry starch amount. The application is here done with smooth rods, instead of conventionally used grooved rods, up to 30% and starch amounts of approximately 1.5–2.0 g/m<sup>2</sup>/side.

The results from the study show a negligible penetration of starch with solids content range from 21 to 30 %. With the low starch penetration, a high surface strength was reached. Additionally, the air permeability of the paper and oil absorption are considerably reduced since the majority of the starch remains on the surface.

However, despite of overall decrease of penetration, local starch penetration of starch can be seen with low viscosity starch even with the highest starch solids content levels. Here, one can suggest, that this local starch penetration into the larger pores of the sheet may be an explanation to high internal strength achieved with low viscosity starch. The high viscosity starch seemed to slightly enhance the starch concentration to the paper surface. This observation was supported by negligible increase of Scott bond values compared to base paper.

It was also seen, that with the low viscosity starch, the runnability of the MSP unit was stable up to starch solids content of 30%. At a high starch viscosity, the web release was stable even up to solids content of 24%. With the high viscosity starch above 24 %, the decreased polymer mobility increased the tension required release the web from the MSP roll.

## 5. Concluding remarks

According to the results presented, it is possible to further decrease energy consumption and investment cost for the after-drying section, compared to starch solids content levels of 8–18 %. Also, the versatility of afterdryer concept can be further enlarged at a broader starch amount range. Also, the previously reported potential increase of efficiency due to decreased wetting of the sheet can be enhanced.

By increasing the solids content of the starch solution to above 20 %, there is less starch penetrating and more starch remaining on the paper surface. The total dry starch amount applied on the paper surfaces was approximately from 3 g/m<sup>2</sup> to 4 g/m<sup>2</sup>, where both starches remained on the surface at solids contents between 21 and 30 %.

However, the development of the internal strength and the visual evaluation of the cross sections may suggest that low viscosity starch could locally penetrate to the larger pores of the sheet despite of the high solids content of the starch. Then, the internal strength of the sheet can be increased also using starch solutions at a very high solids content. Here, the mechanism behind the increased internal strength could be further investigated. The development of internal strength even at very high starch solids values opens new possibilities in optimizing the final paper properties.

The optimization potential for a balance between the molecular weight and the functionality of the starch was demonstrated. According to the results and pilot experience presented, a suitable combination of molecular weight and certain functional properties can give starch both desired stability, runnability and film forming properties as well as provide ability to promote desired paper properties even at very high solids contents.

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