Effects of Hydrophobic Sizing on Paper Dry and Wet-Strength Properties: A Comparative Study between AKD Sizing of NBSK Handsheets and Rosin Sizing of CTMP Handsheets

Antti Korpela,* Aayush Kumar Jaiswal, and Jaakko Asikainen

Alkyl ketene dimer (AKD) and rosin sizing are used in papermaking to decrease paper's tendency to absorb liquid water. Earlier information regarding the effects of internal sizing on paper dry strength is not consistent. In the present laboratory study, AKD sizing of handsheets made from Nordic bleached softwood pulp (NBSK), and rosin sizing of handsheets made from Nordic hardwood chemi-thermomechanical pulp (CTMP) were done *via* an internal sizing method, and by immersing handsheets in aqueous sizing agent dispersion. In the study, AKD sizing had no significant effect on the dry strength of NBSK handsheets. The result corresponds to practical experiences of papermakers. However, both AKD sizing methods resulted in a substantial and long-lasting increase of handsheet wet-strength. Unlike internal AKD sizing of NBSK handsheets, rosin internal sizing of CTMP handsheets resulted in decreased handsheet dry strength. The decrease indicates that, under the conditions present during the experiment, rosin sizing agents interfered with interfiber hydrogen bonding of CTMP fibers. Given that, in practice, no such undesired effects have been commonly linked to rosin sizing, the observed effect may be specific to sheet-making conditions. However, the effect of rosin sizing on strength properties and their variation cannot be ruled out completely.

Keywords: Alkyl ketene dimer, AKD, Rosin, Sizing, Mechanical properties, Wet-strength, PAE

*Contact information: VTT - Technical Research Centre of Finland, Tietotie 4E, P.O. Box 1000, FI-02044 Espoo, Finland; * Corresponding author: antti.korpela@vtt.fi*

INTRODUCTION

Alkyl ketene dimer (AKD) and rosin sizing agents are commonly used in paper making in order to retard absorption of liquid water by paper. Above all, retardation of liquid water absorption is used for improvement of paper coatability and printability, and to make liquid packaging papers water repellent. In so-called 'internal sizing', sizing agents are mixed in papermaking furnish prior to paper web formation. In surface sizing, the application is done on paper using size presses. In both cases, the sizing agents are supposed to attach to fiber surfaces so that their hydrophobic part is orientated outwards from the fibers, thus rendering inherently hydrophilic fiber surfaces more hydrophobic (Strazdins 1989; Cates *et al.* 1989; Scott 1996; Bajpai 2005; Hubbe 2006).

According to common understanding, AKD molecules are capable of forming beta-keto ester bonds with hydroxyl groups of the papermaking fibers. Unbound AKD molecules are supposed to spread on fibers during the drying of paper by surface diffusion and by vapor phase diffusion (Seppänen 2007; Lindström and Larson 2008). Rosin molecules, which are mostly resin acids and modified resin acids, are attached on fiber surfaces by ionic bonds usually *via* alum (aluminum sulphate) or polyaluminum chloride (PAC) (Strazdins 1989; Scott 1996; Bajpai 2005; Hubbe 2006). As introduced in the review article by Hubbe (2006), internal sizing, in general, does not bring any strength to paper that has been completely soaked in water.

One could anticipate that the absorption of sizing agents on fiber surfaces prior to paper web formation would interfere with inter-fiber bonding *via* prevention of formation on interfiber hydrogen bonds, and thus result in decrease of paper strength. However, the published results regarding effects of internal sizing on paper strength are not consistent. In a laboratory scale study by Varshoei *et al.* (2013) tensile-, burst-, and tear-strength of laboratory paper sheets made from old corrugated containers (OCC) was found to decrease as a result of AKD internal sizing. The authors suggested that a decrease in the paper sheet strength was caused by a reduced number of free hydroxyl groups being available to form inter-fiber hydrogen bonds. Contrary to this, Hubbe (2014) notes that, based on the practical experiences of papermakers, internal sizing by AKD has no noticeable effect on paper strength properties. According to Hubbe (2014), a possible reason for the negligible effect is that, under mill conditions, dispersed AKD droplets do not attach to fiber surfaces until after the formation of fiber-to-fiber contacts and interfiber bonds. Also, as Hubbe (2014) points out, a natural supposition is that during the process of drying of paper, spreading of the attached AKD droplets occurs primarily on the non-bonded fiber surfaces. Just as in the case of AKD sizing, reported effects of rosin internal sizing on paper strength properties are also somewhat conflicting. Nada *et al.* (2002) reported decrease of the tensile strength and burst strength of laboratory paper sheets as a result of rosin sizing. In a complete contrast to this, Karademir *et al.* 2007 observed a clear improving effect of rosin sizing on the strength properties of laboratory paper sheets made from various grades of waste paper. In a study by Rahmania *et al.* (2016), the effect of rosin sizing on the strength properties of laboratory paper sheets made from OCC was minor.

The present study aimed to gain further information on the effects of AKD and rosin sizing on both the dry- and wet-strength of paper. Following common industrial practices, NBSK handsheets were sized by AKD, and CTMP handsheets by rosin. CTMP base papers are often sized using rosin owing to a lower price. The sizing experiments were done in a laboratory using two different methods. In the first case simulating ordinary internal sizing, the sizing agents were added into pulp suspension followed by laboratory sheet making and curing of the sheets at an elevated temperature. In the other method, the laboratory paper sheets were immersed in water dispersions of the sizing agent. After the immersion, the sheets were dried and cured again. An assumption was that in the case of immersion method, sizing agents have fewer opportunities to interpose between fibers and thus interfere in interfiber hydrogen bonding and paper strength. In order to enable immersion of laboratory paper sheets, and to get more comparable results, the wet-strength agent polyamide-epichlorohydrin (PAE) was added in pulp suspensions in all trials.

EXPERIMENTAL

Materials

Bleached softwood sulphate pulp (NBSK) sheets and bleached hardwood chemi-thermomechanical pulp (CTMP) sheets were obtained from Finnish pulp mills. The used wet-strength agent polyamine-epichlorohydrin (PAE), cationic alkylketene dimer dispersion (AKD), and cationic rosin dispersion were technical-grade products used industrially in papermaking. According to information from the chemical supplier, both AKD and rosin dispersion contains cationic starch as a dispersing agent (dispersion stabilizer). The used rosin sizing agent also contains polyaluminum chloride (PAC) for anchoring of the rosin on fiber surfaces. Ion-exchanged water was used for laboratory sheet making and diluting chemicals.

Preparation of laboratory sheets and application of AKD and rosin

Laboratory paper sheets were made using uncirculated ion-exchanged water following ISO 5269-1 (2005). Before sheet making, NBSK-pulp sheets were dispersed in water and refined to °SR-value 23.5 using a Voith LR1 laboratory refiner. The °SR-value was measured according to EN ISO 5267-1 (1999). The CTMP-sheets were disintegrated in hot water prior to sheet making following EN-ISO 5263-3 (2004). After hot disintegration, the CSF (ISO 5267-2 (2001)) of the CTMP pulp was 600 mL. The targeted handsheet grammage was 80 g/m^2 when RH was 50%.

In the case of internal sizing ("Method A"), diluted PAE water solution (0.2 wt.-% in water) was added into pulp suspension followed by the addition of the sizing agent (0 - 1.2 wt.-% of dry fiber basis). PAE dosage was 0.3 wt.-% of dry fiber basis in all cases. The pulp suspension was mixed for 10 seconds after both chemical additions, followed by immediate drainage of the suspension. In the case of AKD and rosin sizing, the pH of the pulp was adjusted to the ranges 6.5 to 7.0 and 6.0 to 6.5, respectively, using diluted HCl and NaOH for pH adjustment. For the curing of PAE and sizing agents, the dried laboratory paper sheets were heated in an oven at 80 °C for 120 min. In "method B", cured laboratory paper sheets were immersed by hand in the sizing agent/water dispersion for 5 seconds. After the immersion, excess dispersion flowing on sheet surfaces was removed using plotting paper. The sheets were then wet-pressed and dried in accordance with ISO 5269-1 (2005). Finally, the sheets were cured again in an oven at 80 °C for 120 min. The utilized sizing methods, A and B, are illustrated in the form of a diagram in Fig. 1.

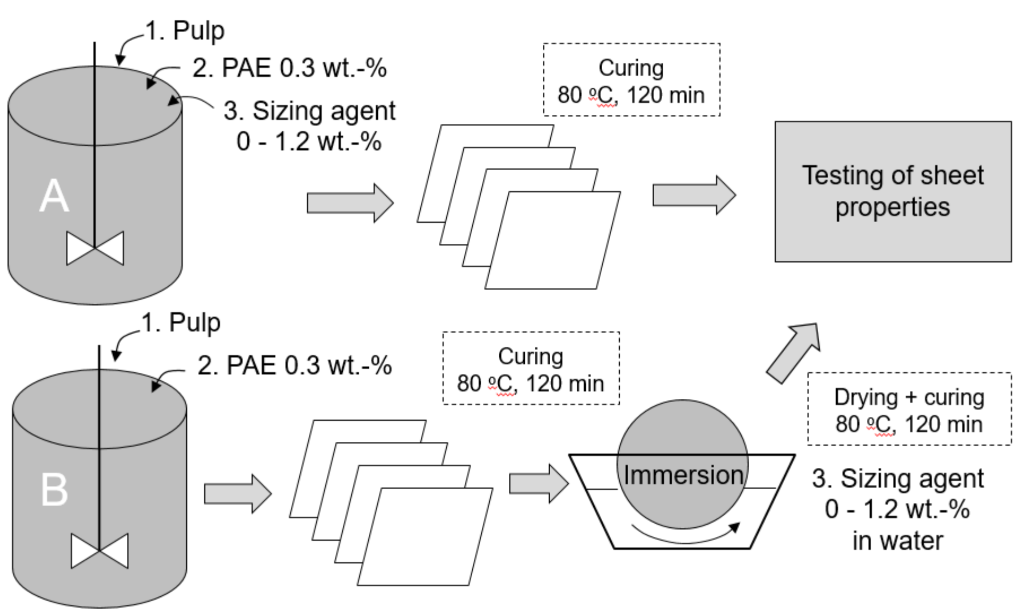

Fig. 1. Sizing of handsheets using internal sizing (Method A) and by immersion of the sheet water dispersion of sizing agent (Method B).

Testing of Handsheet Properties

Laboratory paper sheets were tested according to ISO and TAPPI standards (Table 1). $Cobb_{60}$ values are average values measured on the top and wire sides of the handsheets. In order to get further confirmation about proper functioning of hydrophobic sizing treatments, contact with the angel of water on paper sheets was also monitored. Measurements were carried out using the goniometer Attention Theta Optical Tensiometer (Biolin Scientific, Finland). Measurements were performed at room temperature with distilled water as a probe liquid. Four parallel contact angle measurements were performed at different locations of the wire side of the handsheets and the average value was calculated.

Table 1. Utilized Handsheet Test Methods

Grammage (g/m^2)	ISO 5270 (2012)
Bulk (kg/m^3)	ISO 5270 (2012), ISO 534 (2011)
Tensile Index (Nm/g), Strain at break (%), Elastic modulus, N/mm^2	ISO 5270 (2012), EN ISO 1924-2:2008
Z-dir. tensile strength (kPa)	TAPPI T541 om-99 (1999)
Wet tensile strength (kN/m)	ISO 3781 (2011)
Cobb 60 (g/m^2)	ISO-535 (2014)

RESULTS AND DISCUSSION

Effects of sizing treatments (A and B) on properties of NBSK and CTMP handsheets can be seen in Fig. 2 and in Table 2. The decrease of Cobb-values and the increase of water contact angles indicate the functioning of hydrophobic sizing. As the results show, AKD sizing by way of the internal sizing method (A) or the immersion method (B) had hardly any effect on paper dry strength properties or on bulk. The results are in line with the statements and conclusions of Hubbe (2014). As Hubbe (2014) suggests, AKD internal sizing obviously does not interfere with the formation of interfiber bonds. According to Hubbe (2014), AKD could possibly not attach and spread on fiber surfaces until the formation of interfiber contacts and bonds during formation and drying of paper web. This is evidently also the case when sizing is done by way of immersion of paper sheet in AKD water dispersion

The effects of rosin internal sizing (Method A) on CTMP handsheet properties differed quite substantially from those of AKD sizing of NBSK handsheets (Fig. 2, Table 2). Internal rosin sizing of CTMP handsheets resulted in a considerable decrease of handsheet strength properties, and an increase in bulk. One probable reason for this is that in the performed rosin internal sizing of CTMP handsheets, the rosin droplets were already anchored on the fiber surfaces in the pulp suspension, *i.e.*, prior to dewatering and the formation of interfiber regions of contacts and bonds in paper web. In accordance with this assumption, rosin sizing of CTMP handsheets *via* the immersion method (Method B) resulted in a much smaller decrease of handsheet strength properties. The observed effects of rosin internal sizing are in accordance with findings reported earlier by Nada *et al.* (2002) regarding the effects of internal rosin sizing on the strength properties of various grades of paper. However, it is good to note that papermakers have not widely encountered such undesired effects in commercial paper products. It is quite possible that anchoring of rosin on fiber surfaces depends on papermaking conditions and that the decrease of paper strength is realized particularly in laboratory sheet making conditions. Slightly decreased bulk may be a consequence of the wet-pressing of soaked CTMP handsheets.

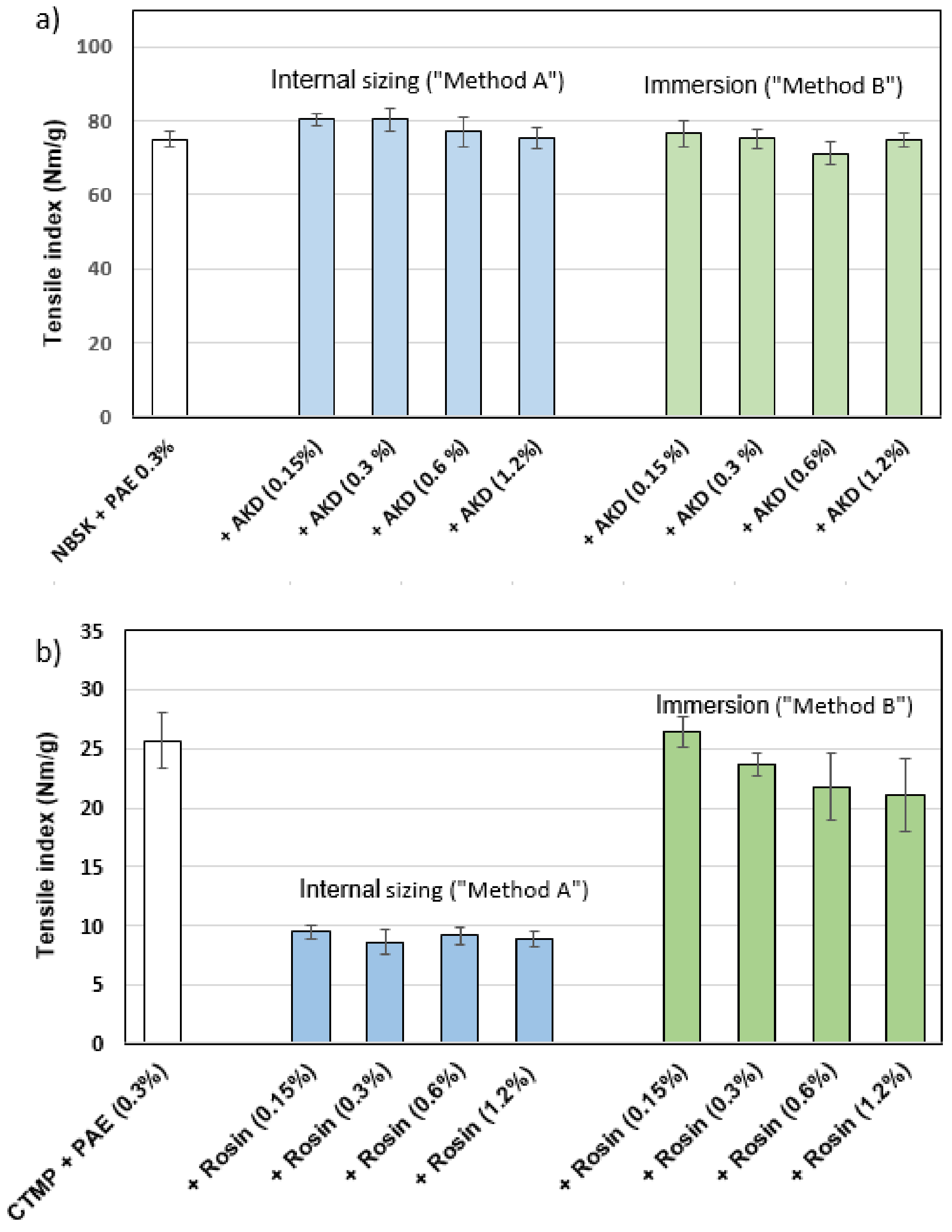

Fig. 2. Effect of internal sizing (Method A) and surface sizing using the soaking method (Method B) by a) AKD and b) rosin on the tensile strength of handsheets made from NBSK and CTMP. In the case of Method A, the dosage (%) indicates the wt.-% of an added sizing agent in pulp suspension on a dry fiber basis. In the case of Method B, the dosage (%) indicates concentration (wt.-%) of the sizing agents in water dispersion used for the soaking of the handsheets.

Table 2. The Effect of Hydrophobic Sizing on Handsheet Strength, Bulk, and Water Repellency

	Z-direct. Tensile Strength (kPa)	Modulus of Elasticity (N/mm^2)	Strain at Break (%)	Bulk (cm^3/g)	Water Absorption $Cobb_{60}$ (g/m^2)	Water Contact Angle ($Ө_{5\ sec}$)
AKD Sizing of NBSK by Method A						
Ref. NBSK + PAE 0.3%	601 ± 38	3442 ± 67	4.9 ± 0.2	1.56 ± 0.02	213 ± 15	< 10
+ AKD 0.15%	640 ± 38	3413 ± 66	5.5 ± 0.2	1.54 ± 0.02	21.1 ± 4.6	124
+ AKD 0.3%	651 ± 31	3391 ± 97	5.4 ± 0.3	1.54 ± 0.01	19.2 ± 2.7	134
+ AKD 0.6%	634 ± 25	3305 ± 99	5.3 ± 0.4	1.54 ± 0.02	18.5 ± 2.5	136
+ AKD 1.2%	613 ± 39	3382 ± 73	5.3 ± 0.3	1.51 ± 0.01	21.0 ± 6.1	138
AKD Sizing of NBSK by Method B						
+ AKD 0.15%	555 ± 38	3770 ± 85	4.4 ± 0.3	1.50 ± 0.02	18.8 ± 4.3	135
+ AKD 0.3%	573 ± 62	3675 ± 226	4.5 ± 0.6	1.47 ± 0.03	18.4 ± 0.7	138
+ AKD 0.6%	570 ± 36	3417 ± 152	5.0 ± 0.5	1.48 ± 0.02	16.9 ± 0.5	137
+ AKD 1.2%	586 ± 31	3547 ± 99	4.9 ± 0.7	1.51 ± 0.01	15.5 ± 0.8	139
Rosin Sizing of CTMP by Method A						
Ref. CTMP + PAE 0.3%	217 ± 19	1435 ± 85	1.4 ± 0.2	2.39 ± 0.07	305 ± 19	< 10
+ Rosin 0.15%	95 ± 4	503 ±26	0.8 ± 0.1	3.68 ± 0.04	347 ± 29	< 10
+ Rosin 0.3 %	75 ± 8	424 ± 35	0.8 ± 0.1	3.73 ± 0.04	140 ± 21	127
+ Rosin 0.6%	91 ± 10	427 ± 41	0.9 ± 0.1	3.63 ± 0.07	59.9 ± 9.3	128
+ Rosin 1.2%	76 ± 6	405 ± 30	0.9 ± 0.1	3,75 ± 0.04	16.2 ± 2.6	139
Rosin Sizing of CTMP by Method B						
+ Rosin 0.15%	251 ± 10	1649 ± 53	1.8 ± 0.1	1.91 ± 0.03	30.8 ± 7.4	129
+ Rosin 0.3%	220 ± 10	1523 ± 36	1.7 ± 0.2	1.95 ± 0.02	19.6 ± 1.5	128
+ Rosin 0.6%	231 ± 18	1515 ± 90	1.4 ± 0.3	1.94 ± 0.04	18.4 ± 1.2	132
+ Rosin 1.2%	228 ± 15	1480 ± 56	1.4 ± 0.4	1.93 ± 0.03	16.1 ± 1.4	126

The “%” indicates the amount of chemical additions in pulp suspension and in soaking liquor (see Fig. 1).

In his article, Hubbe (2006) remarks that hydrophobic sizing of paper has very little effect on the strength of completely soaked paper. According to the results of the present study, AKD internal sizing (Method A) especially had an improving effect on the paper wet-strength (Table 3, Fig. 3a). The improvement was notable even after one week of water soaking the laboratory sheets. As Fig. 3a-b, and 4a-c show, wet-strength to the sized sheets measured after 1, 100, and 10,000 minutes (one week) seem to correlate with the ratio of water to dry solid content of the handsheets. This was the case especially with AKD-sized handsheets. The capability of sizing to improve the wet-strength of the handsheets is obviously linked to the capability of sizing to retard paper and fiber wetting by water. According to general understanding, PAE molecules increase paper wet-strength by forming self-crosslinked, fiber-bond networks repressing fiber swelling and thus protecting fiber-to-fiber bonds when paper is dampened by water (Häggkvist *et al.* 1998; Ozaki *et al.* 2006; Siqueira 2012). The results of the present study indicate that hydrophobic sizing boosts the effect of PAE. The actual mechanism of the effect is still unclear.

In the case of rosin internal sizing of CTMP, the effect of sizing on the wet-strength of the handsheets was not consistent. Unfortunately, the reason for this remained unclear. Improvement of the effect of rosin sizing by way of the immersion method (B) on the wet-strength of CTMP handsheets is quite clear (Table 3, Fig 4d). Overall, the results show that the sizing agents, at least in the utilized sizing conditions, are capable of retarding the absorption of liquid water in paper longer than a week, and that sizing may have a long-term improving effect on paper wet-strength.

Table 3. The Effect of Hydrophobic Sizing on Handsheet Wet-strength (Nm/g) and Dry Substance Content (DS) (wt.-%)

	Wet Tensile Strength Index (Nm/g) and Dry Substance Content (wt.-%)					
	1 min.		**100 min.**		**10^4 min (~1 week)**	
	Nm/g	DS	Nm/g	DS	Nm/g	DS
AKD Sizing of NBSK by Method A						
NBSK + PAE 0.3%	8.51 ±0.65	46.7	7.58 ±0.17	42.2	7.01 ±0.31	39.8
+ AKD 0.15%	28.5 ±1.9	70.3	19.8 ±0.5	61.3	16.6 ±0.6	58.4
+ AKD 0.3%	27.5 ±1.4	70.7	18.5 ±1.4	62.3	15.9 ±1.0	59.0
+ AKD 0.6%	28.9 ±2.5	71.6	22.2 ±0.4	64.3	18.6 ±0.6	61.7
+ AKD 1.2%	31.2 ±2.0	74.3	21.8 ±1.8	63.5	17.2 ±1.5	63.0
AKD Sizing of NBSK by Method B						
+ AKD 0.15%	22.4 ±2.5	72.4	13.8 ±1.4	65.2	11.4 ±0.3	61.9
+ AKD 0.3%	22.8 ±1.5	72.1	13.4 ±0.9	64.0	11.7 ±0.9	61.0
+ AKD 0.6%	24.1 ±3.0	73.6	12.4 ±0.7	63.7	11.0 ±0.7	61.4
+ AKD 1.2%	20.8 ±2.5	66.0	11.8 ±0.5	63.5	11.3 ±1.3	62.7
Rosin Sizing of CTMP by Method A						
CTMP + PAE 0.3%	2.6 ± 0.1	33.5	2.2 ± 0.1	26.7	2.34 ±0.16	27.2
+ Rosin 0.15%	1.4 ± 0.0	27.5	1.4 ± 0.1	22.3	1.21 ±0.07	21.6
+ Rosin 0.3 %	2.3 ± 0.2	47.5	2.0 ± 0.1	22.8	1.80 ±0.14	20.1
+ Rosin 0.6%	2.8 ± 0.3	35.1	2.7 ± 0.1	24.3	2.56 ±0.11	21.3
+ Rosin 1.2%	2.8 ± 0.2	64.7	2.3 ± 0.2	40.3	2.13 ±0.06	34.9
Rosin Sizing of CTMP by Method B						
+ Rosin 0.15%	3.00 ±0.15	48.3	2.8 ± 0.2	41.4	2.9 ± 0.1	39.9
+ Rosin 0.3%	3.91 ±0.42	73.2	2.8 ± 0.1	43.9	2.8 ± 0.1	42.6
+ Rosin 0.6%	4.00 ±0.27	62.2	3.4 ± 0.1	49.6	3.4 ± 0.1	48.6
+ Rosin 1.2%	4.96 ±0.51	66.9	4.0 ± 0.2	51.4	3.9 ± 0.1	52.2

The “%” indicates the amount of chemical additions in pulp suspension and in soaking liquor (see Fig. 1).

The results of the present study supplements earlier reported studies on effects of hydrophobic sizing on paper strength properties. The results also showed that AKD and rosin sizing may increase the wet-tensile strength of the handsheets substantially and that the effect may last well over one week of water soaking time. The repulpability and recyclability of such wet-strength papers would be an interesting topic for further studies.

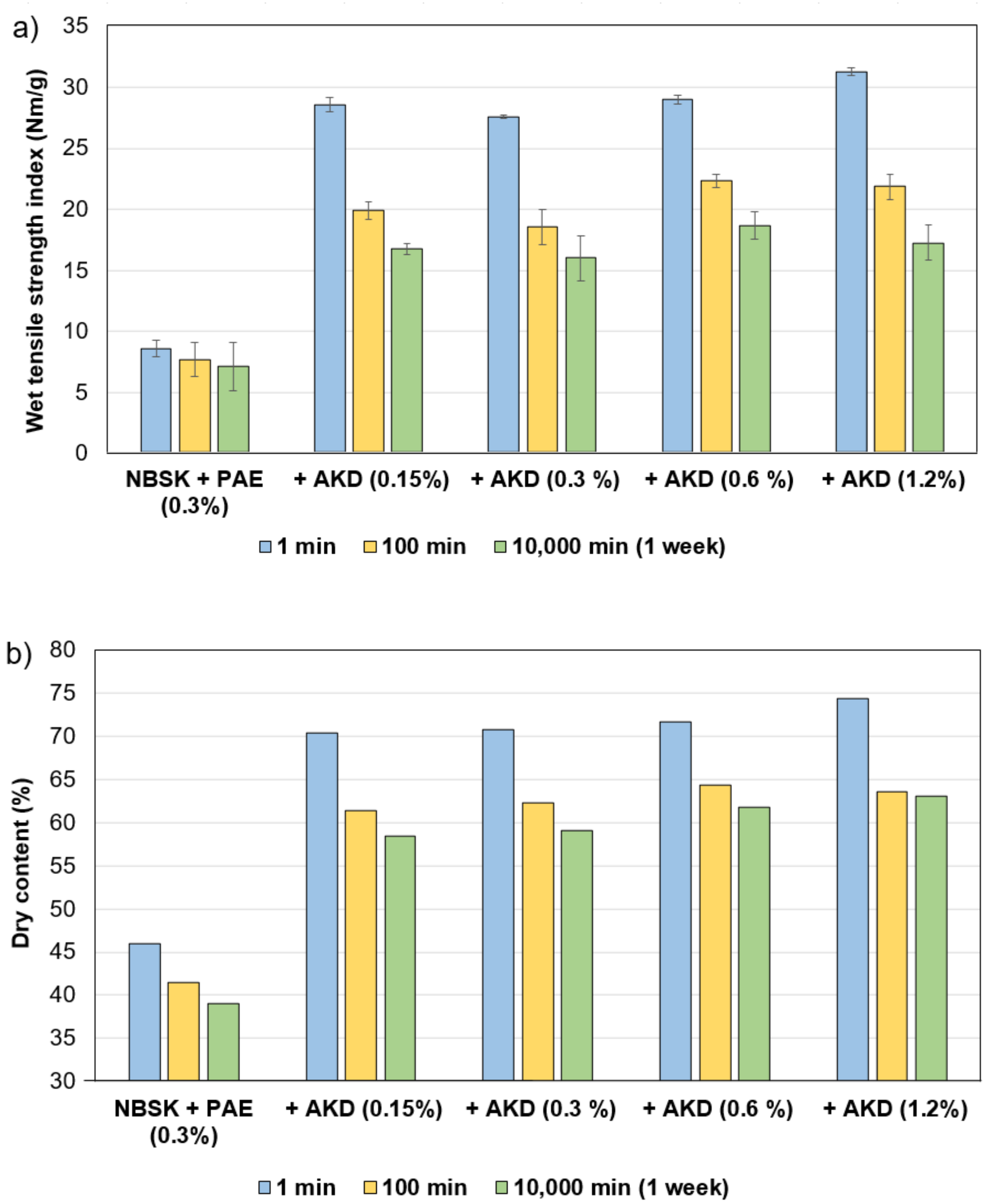

Fig. 3. Effect of internal sizing using AKD on a) wet-strength and b) dry matter content of NBSK handsheets. Variables are AKD dosage (%) and soaking time in water (min). Note: The dry tensile strength of NBSK handsheets with PAE 0.3% was 75.0 Nm/g

It is likely so that the effects of sizing on paper strength properties actually depend on numerous factors, the relative significance of which are not yet fully understood. One such factor is the effect of dispersing agents on the properties of sized papers. Dispersing agents are used for stabilization of the dispersions of commercial sizing agents. They are typically cationic polymeric agents such as cationic starch and cationic synthetic polymers

(Scott 1996; Hubbe 2006). It is quite possible that such polymers also have an effect on the strength and water absorption properties of sized papers. Overall, further information on different factors that affect the properties of sized papers would help papermakers develop manufacturing methods and better performing paper products. Especially for papermakers, it would be useful to get more information to aid in the manufacture of highly water-tolerant yet repulpable and recyclable paper for packaging applications.

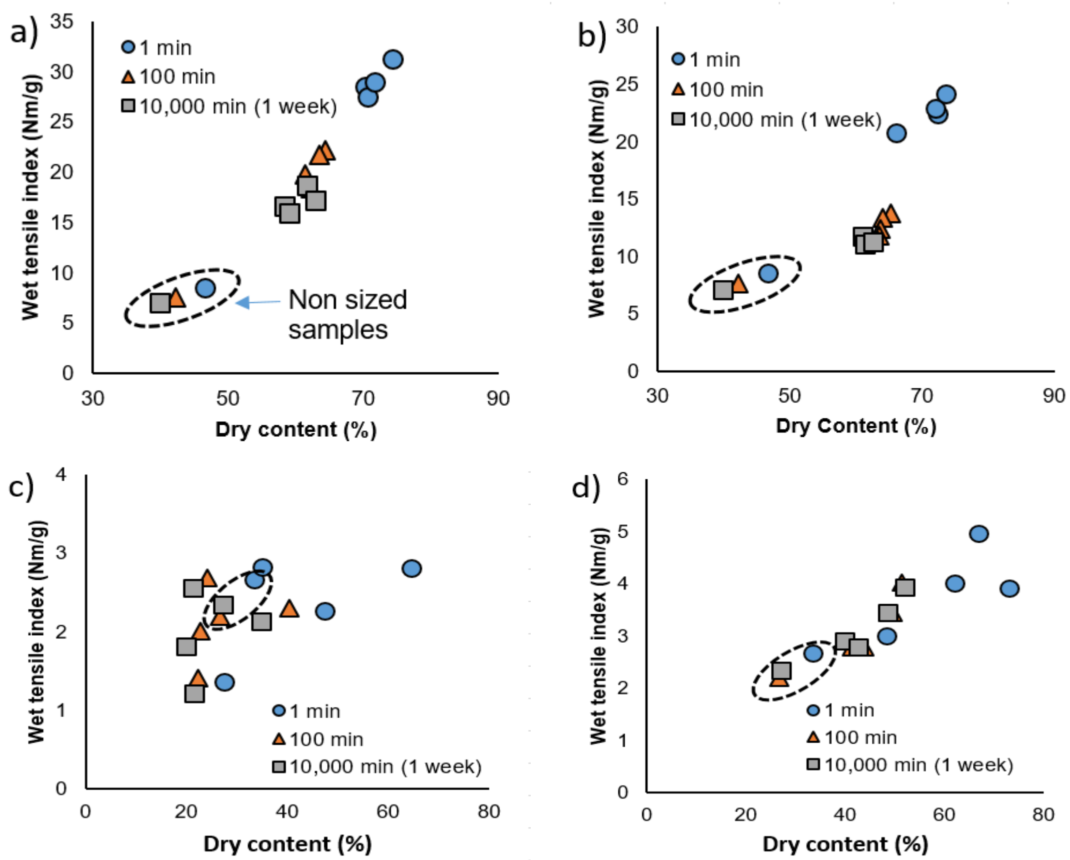

Fig. 4. Relationship of wet tensile index (Nm/g) and dry content (%) of AKD and rosin-sized handsheets. Legends show immersion time of the sheets in water. a) NBSK; AKD sizing using method A. b) NBSK; AKD sizing using method B. c) CTMP; rosin sizing using method A. d) CTMP; rosin sizing using method B.

CONCLUSIONS

1. Sizing of wet-strengthened northern bleached softwood kraft (NBSK) paper with alkylketene dimer (AKD) did not affect the strength and bulk of dry paper. This was the case both in AKD internal sizing and sizing using the immersion method. The results suggest that in internal sizing, AKD does not attach and spread on fiber surfaces until the formation of interfiber contacts and bonds during the formation and drying of paper web. This is evidently also the case as sizing is done by immersion of paper sheet in AKD water dispersion.

2. Rosin internal sizing of wet-strengthened chemi-thermomechanical pulp (CTMP) paper resulted in a decrease in paper dry strength and an increase of paper bulk. It is possible that in the performed rosin internal sizing, rosin droplets were already anchored on the CTMP fiber surfaces in the pulp suspension, *i.e*., prior to dewatering and formation of interfiber regions of contacts and bonds in paper web. In accordance with this assumption, rosin sizing of CTMP handsheets by immersion method (Method B) resulted in a much smaller decrease in tensile strength and z-directional tensile strength.
3. AKD sizing of NBSK handsheets resulted in an increase of paper wet tensile strength. The increasing effect was notable even after 1 week soaking of paper in water. The results indicate that hydrophobic sizing boosts the effect of the polyamido-amine epichlorohydrin (PAE) wet-strength agent. The measured wet-strength correlated with a higher ratio of water relative to dry content of soaked sheets indicating, that AKD is capable of retarding wetting and breakage of interfiber bonds by water.
4. Just like AKD sizing, rosin sizing using the soaking method improved the wet tensile strength of the paper. The improving effect correlated with an increase of paper dry content after soaking it in water. In the case of rosin internal sizing the results were incoherent.
5. Improving effect of AKD and rosin sizing on wet tensile strength of PAE containing paper may offer a way to manufacture of high wet-strength papers. Repulpability and recyclability of such a wet-strength of paper would be an interesting topic for further studies.

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