

Piloting Wet Tensile Strength Development of Polyamide-Amine-Epichlorohydrin Wet Strengthened Nordic Bleached Softwood Pulp Paper by Alkyl Ketene Dimer Internal Sizing

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The results of recent laboratory-scale studies regarding the joint effects of wet strengthening agent polyamide-amine-epichlorohydrin (PAE) and internal alkylketene dimer (AKD) sizing on Nordic bleached softwood pulp (NBSK) handsheets indicate that AKD internal sizing increases the wet strength of PAE wet strengthened handsheets. The boosting effect of the internal AKD sizing was long-lasting. At the molecular level the actual mechanism behind the effect is unclear. The present study examines if the enhanced effect of internal AKD sizing, which is potentially exploitable in practice, also occurs in paper made by VTT's Suora pilot paper machine. The trials were done using a similar type of NBSK pulp and the same chemicals as in the laboratory-scale studies. The paper was formed at 400 m/min using a hybrid dewatering mode (Fourdrinier + gap). Just as in the laboratory-scale studies, the internal AKD sizing of PAE wet-strengthened NBSK paper resulted in a long-lasting increase of the paper wet strength. In accordance with the laboratory trials, internal AKD sizing had no noticeable effect on the dry tensile strength of the pilot-made paper. The only apparent difference to the earlier observations was an increase of dry tensile strength of the paper treated solely with PAE.

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INTRODUCTION

A disadvantage of high dosing with the polyamide-amine-epichlorohydrin (PAE) wet-strength agent in papermaking is that it can make repulping of broke and wastepaper difficult (Siqueira 2012; Su *et al.* 2012). The PAE may also contribute to paper machine felt filling and thus impair the paper machine runnability and performance (Ringold and Fuhrman 2019). Because of these side-effects and for economic reasons, papermakers often try to minimize the use of PAE. Recent laboratory studies showed that the wet tensile strength of handsheets that are wet-strengthened by PAE can be substantially increased by internal alkyl ketene dimer (AKD) sizing. According to the laboratory trials, the improving effect of the AKD internal sizing is long-lasting, and it is most notable with a relatively low or moderate added amount of PAE. Although internal AKD sizing was observed to increase the paper wet-strength, no evident sign of worsened repulpability came up in the comparative repulpability tests. Overall, the results of the laboratory-scale studies suggest that the use of smaller amounts of PAE together with AKD could be a feasible option for paper mills facing repulpability or paper machine runnability problems related to the high usage of PAE.

The molecular-level mechanism behind the observed synergistic effect of PAE and AKD on the wet strength of the paper is not clear. According to general understanding, PAE, which is a cationic water-soluble polymer, increases the paper wet strength by forming self-cross-linked fiber bond polymer networks in the paper. The bonds with fiber carboxyl groups are suggested to be covalent and water insensitive by nature (Häggkvist *et al.* 1998; Ozaki *et al.* 2006; Obokata and Isogai 2007; Siqueira 2012). The AKD molecules are thought to make paper more hydrophobic by spreading and attaching on fibers so that the hydrophobic aliphatic part of the AKD molecule is oriented outwards from the fibers and the reactive diketene group towards the fibers. The diketene groups are believed to form covalent beta-keto ester bonds with the hydroxyl groups of the papermaking fibers (Cates *et al.* 1989; Strazdins 1989; Scott 1996; Bajpai 2005; Seppänen 2007; Lindström and Larson 2008; Hubbe 2006, 2014). In the preceding laboratory-scale studies, PAE was added first into the pulp suspension followed by the AKD sizing agent. In principle, the interaction of the added PAE and AKD may affect the retention of PAE and AKD, or cause changes in the way they are bonded and spread on the fibers. Hagiopol and Johnston (2012) suggest that AKD may react with PAE resin to make a hydrophobic cationic polymer (β -ketoamide). The preceding laboratory studies showed that the wet strength of water-soaked handsheets containing both PAE and AKD correlated with the moisture content of the tested handsheets. The correlation was clear especially for handsheets containing low or moderate added amounts of PAE (0.15 wt% and 0.45 wt%). According to a study by Häggkvist *et al.* (1998), PAE is efficient at reducing the water-induced swelling of the fiber wall of unbleached kraft pulp. Siqueira (2012) notes that PAE resins may slightly reduce the water absorption capacity of paper, which is useful in packaging products but not in tissue papers. The observed positive correlation of the water-soaked paper dry content and wet strength (Korpela *et al.* 2021, 2022) indicate that AKD is capable, one way or another, of boosting the decreasing effect of PAE on the paper water absorption capacity.

Another observation from previous laboratory studies was that the AKD internal sizing had very little if any effect on the dry tensile strength and density of PAE wet-strengthened handsheets (Korpela *et al.* 2021, 2022). As Hubbe (2014) suggests, AKD internal sizing does not apparently interfere with the formation of inter-fiber bonds. According to Hubbe (2014), dispersed AKD droplets possibly do not attach and spread on the fiber surfaces until the formation of inter-fiber contacts and bonds during the formation and subsequent drying of the paper web. Thus far, there seem to be no published studies regarding the effect of PAE on the formation of inter-fiber hydrogen bonds or the way dewatering conditions affect the absorption and localization of PAE on and between the fibers.

The present study examined whether the synergistic effect of PAE and AKD on the wet strength of the paper also occurred in pilot-scale papermaking, where especially the dewatering conditions in the wire section differ from those of laboratory sheet making. The idea was to carry out the pilot trials under conditions that are otherwise as close as possible to the laboratory sheet-making conditions (Korpela *et al.* 2021, 2022), and in this way obtain comparable information on the effect of the web forming conditions on the joint effect of PAE and AKD on the paper properties. In the study, a similar type of NBSK pulp, and the same PAE and AKD products were used as in the laboratory study. After the forming and wet pressing sections, the paper samples were dried and tested in the same way as the laboratory handsheets. Instead of using ionic exchanged water, all dilutions of the pilot trials were completed using tap water. In the pilot trials, the maximum water conductivity was around 600 μm , which from the practical papermaking point of view

means low conductivity, which is similar in practice to some mills producing uncoated wood-free paper grades (Xu *et al.* 2017). The effect of such a low water conductivity on the retention and functioning of PAE and AKD is probably minor.

EXPERIMENTAL

Materials

Ready-refined bleached softwood sulphate pulp (NBSK) was obtained from a Finnish paperboard mill. The Schopper-Riegler ($^{\circ}$ SR) value of the delivered pulp measured according to ISO 5267-1 (1999) was 18. The wet strength agent PAE and cationic AKD dispersion for this study were the technical-grade products used in the preceding laboratory trials (Korpela *et al.* 2021, 2022). Both PAE and AKD were used as such in the pilot trials. In the present study “low”, “moderate”, and “high” amounts of PAE refer to added dry-basis amounts of 0.15 wt%, 0.45 wt%, and 1.35 wt%, and for AKD 0.15 wt%, 0.30 wt%, and 0.60 wt% added amounts, respectively. Tap water was used for all dilutions.

Pilot-scale Web Forming

The trials were conducted using VTT Technical Research Centre of Finland’s (VTT’s) pilot-scale paper and board making machine in Jyväskylä, Finland. The pilot machine included an OptiFlo-type headbox, forming and pressing sections, and vacuum equipped sampling rolls for sampling and reeling of wet pressed paper (Lehmonen *et al.* 2019). In the forming unit the accessible paper web speed was max. 2500 m/min and headbox flow rate 240 L/s/m. The web and fabric width were 300 and 500 mm, respectively. The press section consisted of a single nip shoe press with a nip length of 350 mm. Max line load of the press was 2000 kN/m. The web width was approximately 210 mm, and the belt width was 700 mm. The pilot machine can be run in a fourdrinier, hybrid, or gap mode. The current study was performed in the hybrid mode. In the pilot trials the PAE was added into headbox feed flow in front of the screen and the AKD was added to the feed flow at the last insertion point before the headbox. The white water was recirculated. In the study, the web samples were taken after reeling the wet pressed web. The process set-ups and realized values of the pilot trial are shown in Table 1.

Table 1. Process Set-Ups of the Pilot Trials

Headbox	OptiFlo
Former	Hybrid
Machine speed (m/min)	400 (400 to 401)
Jet-to-wire ratio	0.99 (0.99)
Headbox consistency (%)	0.40 (0.35 to 0.40)
pH	8.0 (7.8 to 8.1)
Temperature, ($^{\circ}$ C)	45.0 (41.2 to 46.2)
Wet press, 1 shoe press (kN/m)	1000 (1000 to 1001)
Water conductivity (μ m/cm)	< 500 (10 to 600)
Dryness after wet press (%)	40.0 (39.9 to 41.2)
Target basis weight (g/m^2) (RH 50%)	80 (69 to 80)

Realized values are shown in parenthesis; RH: relative humidity

Drying and Curing of Web Samples, and Testing of Paper Properties

Wet pressed web samples were dried following ISO 5269-1 (2005). For the final curing of the PAE and the AKD, the dried paper sheets were heated in an oven at 80 $^{\circ}$ C for

120 min. The targeted handsheet grammage was 80 g/m² when the RH was 50%. The laboratory paper sheets were tested according to ISO and TAPPI standards (Table 2). The dry strength properties of the papers were measured in both the machine direction (MD) and cross direction (CD) and reported as geometric averages.

The repulpability of the prepared papers was tested using a modified Michelman repulpability test (Fibre Box Association 2013). The paper samples (11 g) were first cut into 3 cm × 10 cm pieces and were conditioned overnight in a standard climate, at 23 °C, 50% RH. This was followed by disintegration of the test pieces without pre-swelling, first using a Waring blender 38BL40 (Waring Commercial, Stamford, CT, USA) at a consistency of 1.7% (o.d.). The water temperature was 52 °C. The pH value was 7 and a disintegration time of 3 times 20 s was applied. After the Waring blender disintegration, the pulp suspension was additionally disintegrated in a standard disintegrator (Lorenzen & Wettre, Kista, Sweden) described in EN ISO 5263 (2004) for 5 min at 52 °C. After this, Somerville screening was done according to TAPPI T275 (R2018) (slotted screen #0.15 mm). The amount of reject (wt%) after screening was finally determined.

Table 2. Utilized Handsheet Test Methods

Grammage (g/m ²)	ISO 5270 (2012)
Density (kg/m ³)	ISO 5270 (2012), ISO 534 (2011)
Tensile Index (Nm/g),	ISO 5270 (2012), EN ISO 1924-2 (2008)
Wet Tensile Strength (kN/m)	ISO 3781 (2011)
Cobb 60 (g/m ²)	ISO-535 (2014)
Repulpability	Fibre Box Association, voluntary standard (2013)

RESULTS AND DISCUSSION

The previous laboratory studies indicated that AKD internal sizing had no noticeable effect on the dry strength of handsheets that were wet strengthened using PAE (Korpela *et al.* 2021, 2022). In the studies, PAE alone had no effect on the dry strength of the handsheets either. Differing from the laboratory studies, the present pilot-scale papermaking study also included trials where papers were internally AKD sized without the usage of PAE. The effect of sole internal AKD sizing and sole PAE on the dry and wet strength of the pilot machine papers are shown in Fig. 1. The results show that the AKD internal sizing alone has no noticeable effect on either the dry or wet strength of the paper. This result is in accordance with the indications of the previous laboratory studies and with the papermakers' practical experience (Hubbe 2014). The results in Fig. 1 show also that PAE increases not only the wet strength of the pilot machine-made paper but this time also its dry strength. The magnitude (Δ) of the increase in the wet and dry tensile strength was of the same size. The increasing effect on the dry tensile strength is in conflict with earlier laboratory studies (Korpela *et al.* 2021, 2022) and results reported by Vähä-Nissi *et al.* (2018). In contrast, several other published studies report that PAE increases not only the paper wet strength but also the dry strength (Espy 1995; Kitaoka and Tanaka 2001; Su *et al.* 2012). The reason for the conflicting results of the preceding laboratory studies and present pilot study is not clear. They may arise from the influence of PAE on the formation uniformity of the handsheets and the pilot-made paper. It is worth noting that in both cases the wet-pressed paper sheets were dried and cured (80 °C for 2 h) and tested in the same

way, *i.e.*, the cause of the conflicting results is probably not in the different PAE curing degrees.

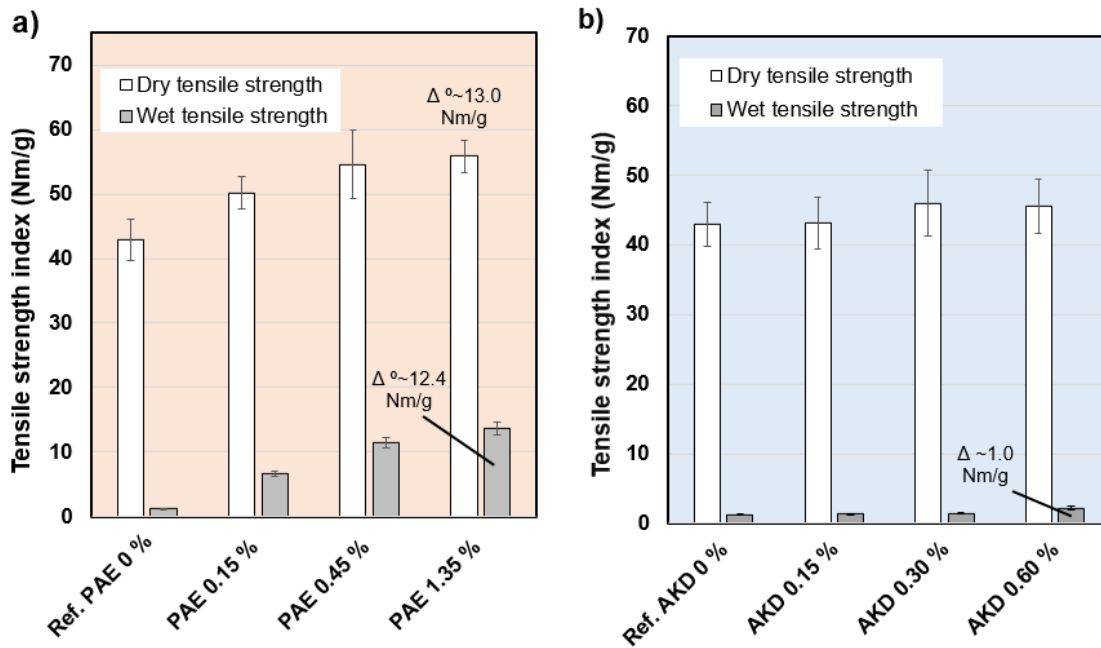


Fig. 1. The effect of PAE (a) and AKD (b) alone on the dry and wet tensile strength (geom. averages) of NBSK paper. The water soaking time was 60 s. The “%” indicates the amounts of chemical additions in the paper furnish. Standard deviations of the measurement are indicated by the error bars.

The joint effect of PAE and internal AKD sizing on the wet strength (60 s soaking time) of the pilot machine-made papers is shown in Fig. 2. The triangles in Fig. 2 refer to the corresponding results of the preceding laboratory-scale study (Korpela 2022). The synergistic effect of PAE and internal AKD sizing is clearly visible in the wet strength for both the handsheet and pilot-made paper. The reason for the somewhat higher wet strength of the handsheets may result from higher retention of PAE and AKD in handsheet making. Unfortunately, due to the laboriousness of the quantitative analysis, the retention of PAE and AKD in the papers could not be quantified in the present study.

In the previous studies, the PAE and internal AKD sizing had a minimal effect on the dry tensile strength of the handsheets (Korpela *et al.* 2021, 2022). The improved effect of PAE on the dry tensile strength (geom. avg.) of the pilot machine-made paper (Fig. 1) is evident also when the paper is sized internally using AKD (Fig. 3). However, the PAE and the AKD sizing had no clear effect on the density of the handsheets and the pilot machine-made paper (Fig. 3). The higher dry tensile strength and higher density of the handsheets may be consequences of the different fiber properties of the pulps used for handsheet making and for pilot machine-made paper, or of higher wet-pressing intensity in the handsheet making. Additionally, the somewhat poorer formation (cloudiness) of the pilot machine-made paper may have influenced the strength properties. According to visual estimations neither PAE or AKD had an effect on the evenness of the pilot machine-made papers. The MD/CD tensile index ratio of the pilot machine-made papers was in the range of 2.2 through 2.7. The addition of PAE or AKD did not have any consistent effect on the paper’s MD/CD dry and wet tensile index ratios.

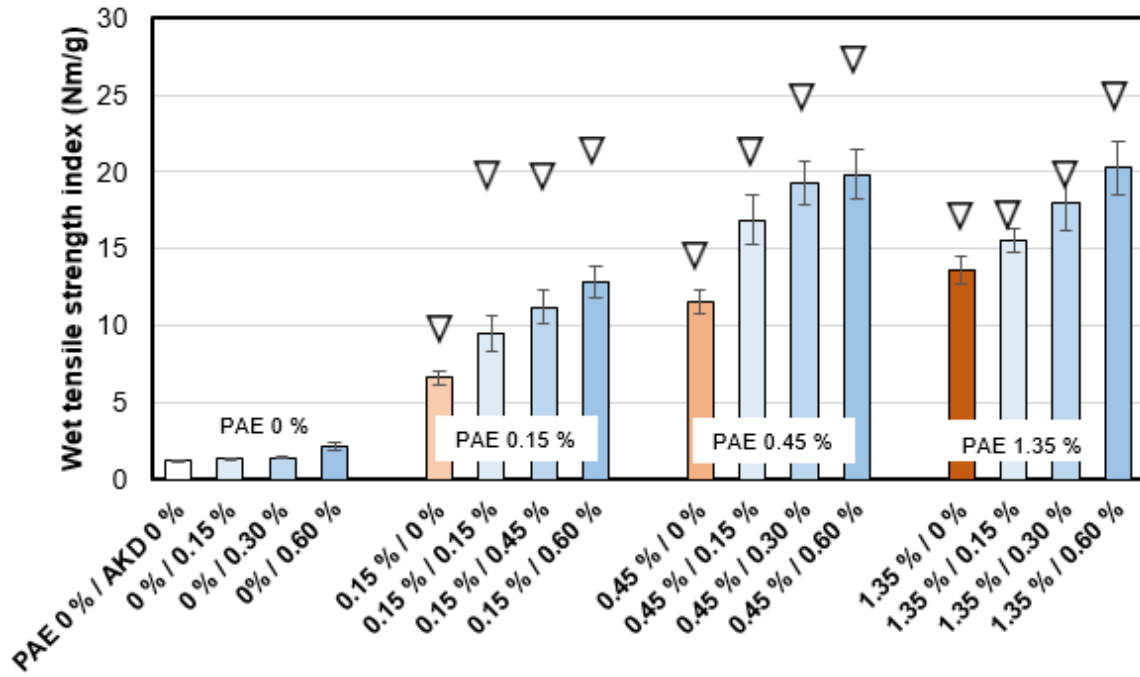


Fig. 2. The effect of AKD internal sizing on the wet tensile strength (geom. average) of the NBSK pilot machine-made paper wet strengthened by PAE. The water soaking time was 60 s. The “%” indicates the amounts of chemical additions in the paper furnish. The triangles refer to the corresponding measurement results of the preceding laboratory study (Korpela *et al.* 2022).

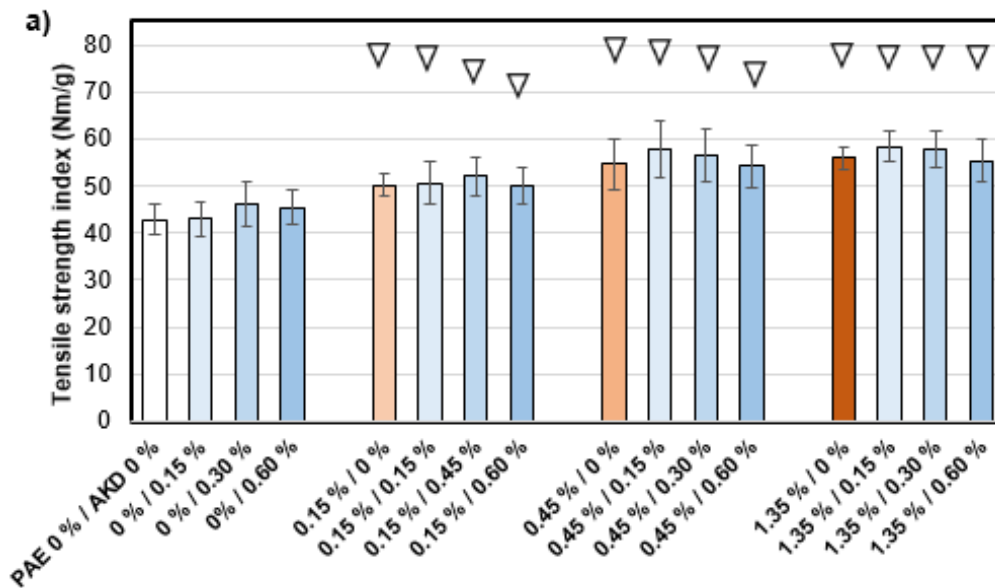


Fig. 3a. The effect of AKD internal sizing on a) the dry tensile strength (geom. Avg.) and b) density of NBSK pilot machine-made paper wet strengthened by PAE. The “%” indicates the amounts of chemical additions in the paper furnish. The triangles refer to the corresponding measurement results of the preceding laboratory study (Korpela *et al.* 2022).

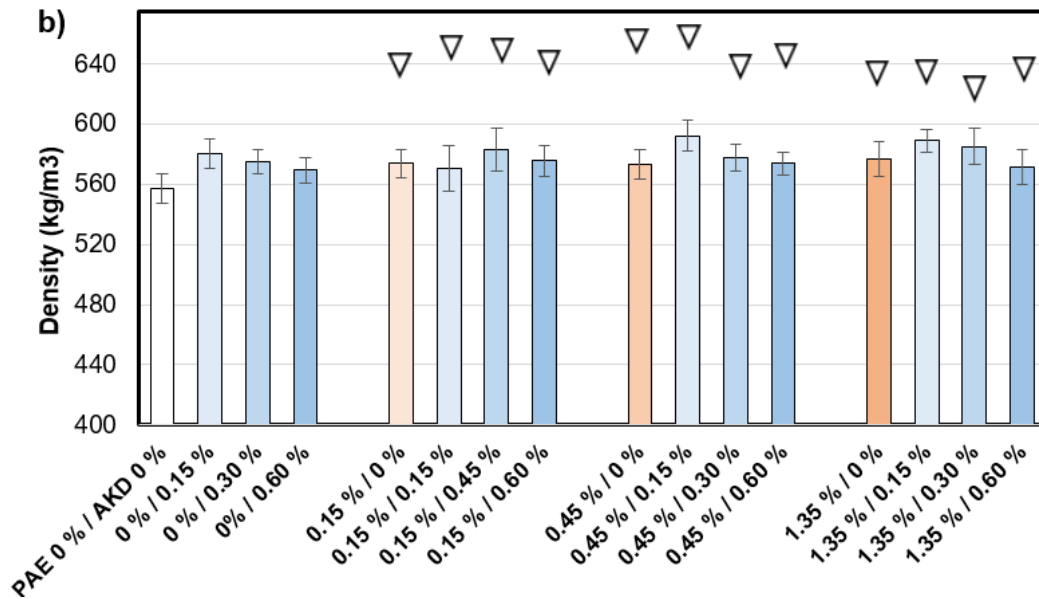


Fig. 3b. The effect of AKD internal sizing on a) the dry tensile strength (geom. Avg.) and b) density of NBSK pilot machine-made paper wet strengthened by PAE. The “%” indicates the amounts of chemical additions in the paper furnish. The triangles refer to the corresponding measurement results of the preceding laboratory study (Korpela *et al.* 2022).

Figure 4 shows the effect of PAE and internal AKD sizing on the water absorption (Cobb₆₀) of the pilot machine-made papers.

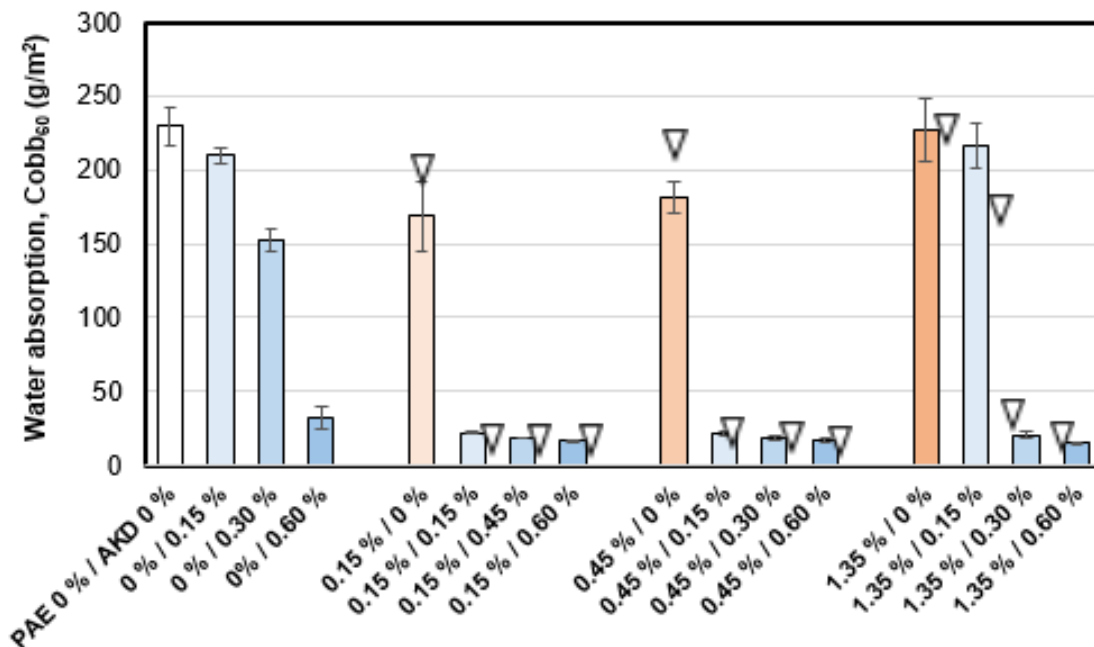


Fig. 4. The effect of AKD internal sizing on the water absorption (Cobb₆₀) of the NBSK pilot machine-made paper wet strengthened by PAE. The Cobb₆₀ values are averages for the top and wire sides. The “%” indicates the amounts of chemical additions in the paper furnish. The triangles refer to the corresponding measurement results of the preceding laboratory study (Korpela *et al.* 2022).

The effects are in accordance with earlier laboratory studies (Korpela *et al.* 2021, 2022). No consistent noticeable differences between the results of Cobb₆₀ measurements made on the paper wire and top side were apparent either. It is worth noting that it seems the PAE and internal AKD sizing has a synergistic decreasing effect also on the paper's water absorption. Additionally, the molecular-level mechanism behind this synergistic effect is not known. It may be related to more efficient spreading of AKD on PAE-covered fiber surfaces, or to chemical reactions of AKD with PAE (Hagiopol and Jonston 2012). The relatively high water absorption of the handsheets and pilot machine-made papers with 1.35% PAE and 0.15% AKD (added amounts) is possibly a consequence of low retention of the cationic charged AKD droplets on the fibers covered by high amounts of cationic charged PAE.

Figure 5 shows the effect of PAE and the AKD sizing on long-term wet strength (MD) of the pilot-made papers. The MD/CD wet tensile index ratio of the papers was in the range of 2.1 to 2.8. The results are in accordance with the earlier laboratory studies. The relative influence of AKD on the wet strength of the pilot machine-made papers was somewhat smaller though. This may be related to unequal retention of either PAE or AKD in the laboratory sheet and pilot paper making. For the laboratory handsheets the wet tensile strength correlates with the dry content of the water-soaked paper sheets (Fig. 6). Additionally, this result is in accordance with the findings of the laboratory study (Korpela *et al.* 2022).

The effect of AKD internal sizing on repulpability of the pilot machine-made paper is shown in Fig. 7. The results of the present study and the laboratory study (Korpela *et al.* 2022) are somewhat different. In the present study, the estimation of paper repulpability is based on the amount of rejected (wt%) material in the final screening of the repulped paper, whereas in the laboratory study, the estimations were based on the number of visible specs in the handsheets after the repulping (TAPPI UM 213 2012). In the laboratory study, just those handsheets that contained AKD and the highest amount of PAE (added amount 1.35 wt%) showed slightly worsened repulpability. In the present study, papers containing a moderate and high amount of PAE (added amount 0.45 wt% and 1.35 wt%, respectively) showed worse repulpability. The result is in accordance with the long-term increase of the paper wet strength. From the repulpability point of view, the use of a low added amount of PAE with internal AKD sizing seems to be the most favorable option. The increase of wet strength from the AKD internal sizing is substantial and long-term, yet not as permanent as the increasing effect of PAE on the wet strength.

The present pilot trials mostly confirmed the result of the preceding laboratory trials. The largest difference in the results concerns the effect of PAE on the dry tensile strength of the handsheets and the pilot machine-made papers. The reason for the observed difference is not clear. Overall, the molecular-level mechanisms behind the synergistic effects of PAE and internal AKD sizing on the water absorbency and wet strength of paper are not known. It is possible that PAE enhances the spreading and orientation of AKD on the fibers in some favorable way resulting in reduced water absorption of the paper. As suggested by Hagiopol and Jonston (2012), AKD may also react covalently with PAE. The chemical reaction of AKD with PAE may result in the formation of a more protective film against water absorption. Comprehension of the mechanism could help to clarify the full potential of the synergistic effect of PAE and AKD and its utilization in practice.

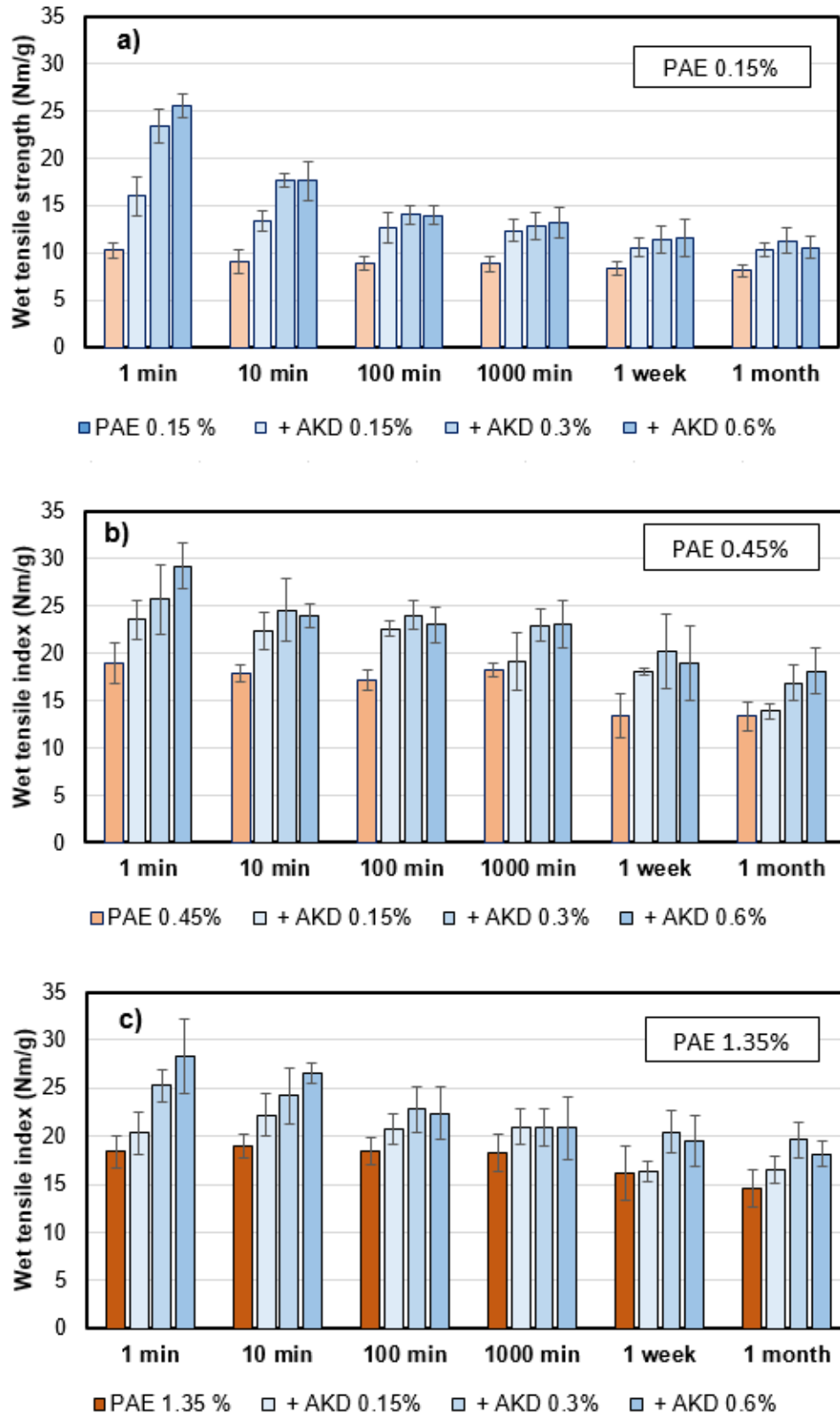


Fig. 5. The effect of AKD internal sizing on the long-term wet tensile strength (geom. Avg.) of NBSK pilot machine-made paper wet strengthened by PAE: a) PAE 0.15%, b) PAE 0.45%, and c) PAE 1.35%. The “%” indicates the amounts of chemical additions in the paper furnish. The wet tensile strength is measured in the MD.

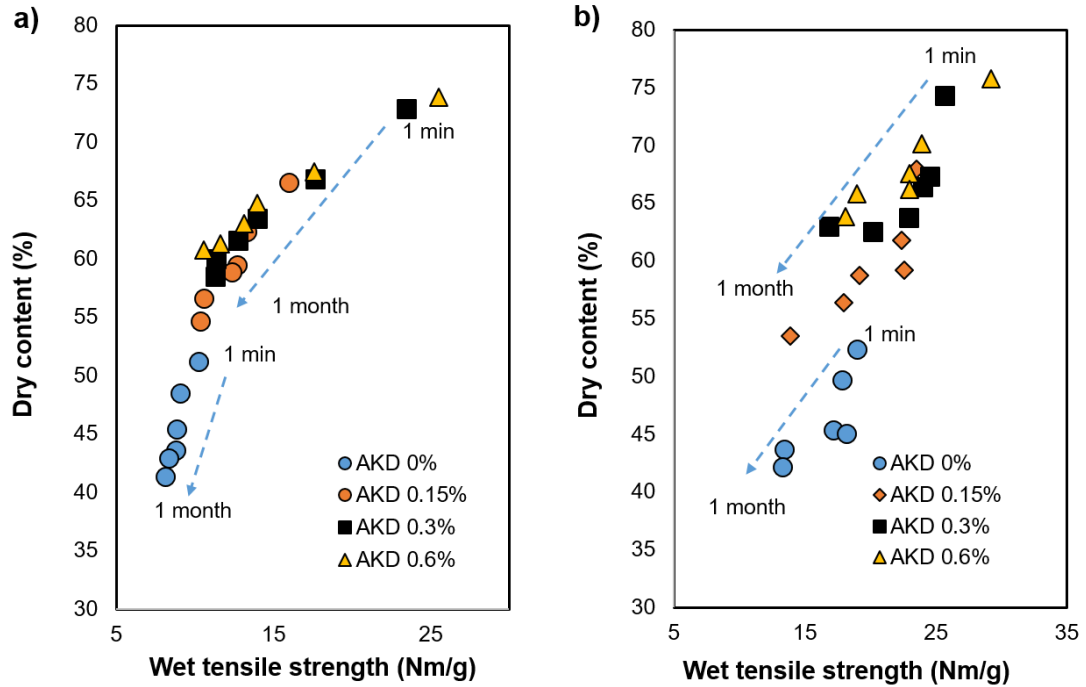


Fig. 6. The relationship of the wet tensile strength index (Nm/g) and dry content (%) of AKD internally-sized NBSK pilot machine-made paper wet strengthened by PAE. The wet strength is measured in the MD. The “%” indicates the amounts of chemical additions in the pulp suspension: a) PAE 0.15%, b) PAE 0.45%.

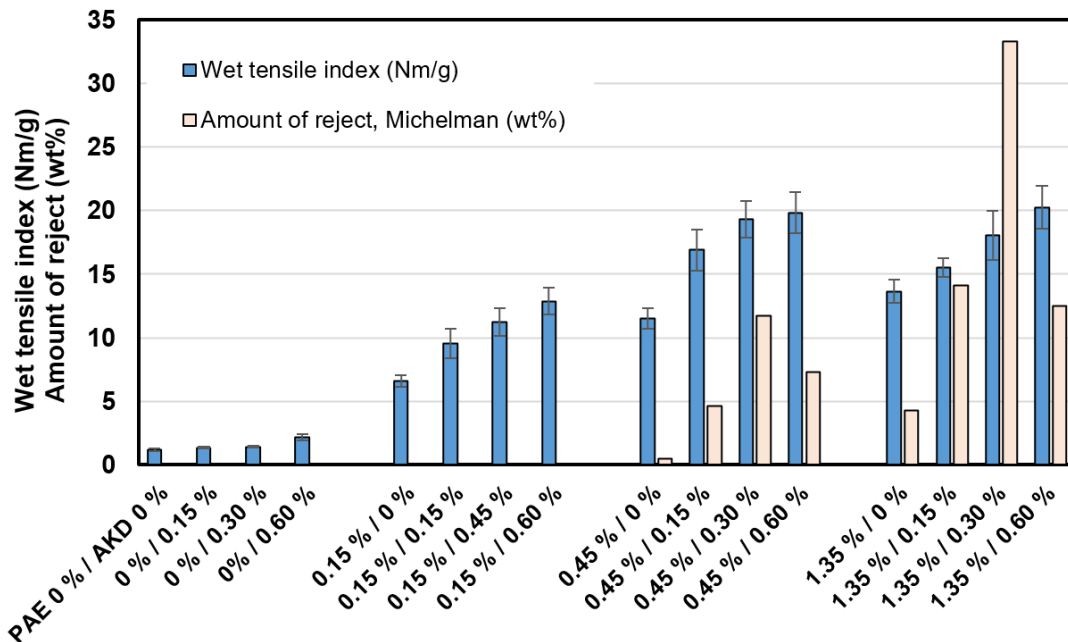


Fig. 7. The effect of AKD internal sizing on the wet tensile strength (geom. avg., soaking time 60 s) and amount of reject (wt%) in a Michelman repulpability test of the NBSK pilot machine-made paper wet strengthened by PAE: The “%” indicates the amounts of chemical additions in the paper furnish.

It is also good to note that the effects of the mill-specific process conditions, such as the process water pH and conductivity, and other papermaking chemicals and constituents, on the functioning and interaction of PAE and AKD are not yet known. Therefore, in experimental laboratory- and pilot-scale works it would be useful to imitate the relevant mill conditions as close as possible.

CONCLUSIONS

1. The results show that alkylketene dimeter (AKD) internal sizing boosts the effect of poly(amidoamine-epichlorohydrin) (PAE) on the wet strength of paper by reducing water absorption of the paper during water soaking. On the other hand, the water absorption is influenced by the boosting effect of PAE on the hydrophobic sizing action of AKD. The synergistic effect of PAE and AKD were both apparent in the pilot machine-made papers and laboratory handsheets.
2. The molecular-level mechanisms behind the observed synergistic effects are not yet understood. It is quite possible that in practice process parameters, such as the pH and conductivity of the process waters, as well as other papermaking constituents, have an effect on the mutual interaction PAE and AKD, and thus on their synergistic action in paper
3. In paper product development and planning of laboratory and pilot trials for paper product development it could be useful to take the possible effects of different papermaking conditions and compositions on functioning and interaction of PAE and AKD into account, and to imitate the relevant mill conditions as far as possible.

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