

# Enhancing Subsequent Kraft Fiber Dewatering Properties by using Fiber Polyamide-epichlorohydrin (PAE) Treatment to Prepare a Dry Pulp Product

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The energy needed for the dewatering and drying of wet paper web represents around half of the energy consumption of papermaking processes. The present work examined whether the dewatering and drying of paper could be enhanced during a previous pulp drying process by pretreating the fibers with polyamide-epichlorohydrin (PAE). According to the hypothesis, the cured PAE restrains swelling and water absorption of water-wetted fibers by forming a fiber-bound, self-crosslinked polymer-network on the fiber surfaces. The hypothesis was tested by adding PAE to never-dried kraft pulp slurry followed by pulp thickening, drying, and final curing of the PAE-resin. After this, the PAE-treated fibers were dispersed in water, and their water retention values (WRV) and Shopper-Riegler values ( $^{\circ}\text{SR}$ ) were measured. The PAE pretreatments notably decreased the fibers' WRV and  $^{\circ}\text{SR}$ , indicating improved water removal of paper web in the paper machine forming and drying section. Compared to chemical crosslinking pretreatments, which also can be used to decrease fibers WRV and  $^{\circ}\text{SR}$ -value, a notable advantage of PAE-pretreatment is milder required curing conditions of the PAE, which makes implementation of the method easier in practice. Due to decreased fiber-to-fiber bonding capability, the PAE-treated specialty fibers could take advantage especially as a bulking aid of paperboard, tissue, and absorbent materials.

DOI: 10.15376/biores.19.3.5227-5238

Keywords: Market pulp; Dewatering; Bulking aid; Energy consumption; PAE; Paper properties

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## INTRODUCTION

As papermaking fibers are mixed with water, the fiber lumens, fiber wall pores, and amorphous parts of the fiber walls become filled and saturated by water. Due to the water absorption, the fibers swell, especially in their cross-direction. Basically, the amount the fibers absorb the water (g/g) increases as the fiber's swelling capacity in water increases (Weise *et al.* 1996; Salmen and Berthold 1997; Hubbe and Heitmann 2007). In papermaking, typically around 150 g of water per 1 g of fiber are removed in the paper machine forming section, 2 g/g in the wet pressing section, and around 1.5 g/g in the drying section, respectively. The water removed in the paper machine forming section is often characterized as "free water", whereas the water evaporated in the drying section is described as "bound water", having attractive forces with cellulosic hydroxyl and carboxyl groups. This water cannot be compressed or centrifuged from the fibers, but it needs to be removed by evaporation. Due to the inter-molecular attractive forces of water molecules and lignocelluloses, evaporation of the bound water from paper web consumes a

substantially high amount of energy (Maloney *et al.* 1998; De Beer *et al.* 1998; Wang *et al.* 2006). In a study by Laurijssen *et al.* (2013), the reported specific drying energy consumptions of graphical papers were in the range of 5.2 to 9.5 GJ/t paper. Depending on the paper grade, the drying energy amounts to approximately 50 to 70% of the total energy consumption of the paper making (Kramer *et al.* 2009; Laurijssen *et al.* 2013; Hubbe 2021).

The drying of kraft pulp results in decreased water absorption and swelling of the fibers in water. Although pulp drying is costly, it is a common practice not only in market pulp mills but also in some integrated mills, as drying of the fibers can result in net savings due to improved water removal and paper machine speed. In general, the tensile strength properties of the dried pulps are inferior to those of never-dried pulps; but in many cases, the achieved improvements in the dewatering outweigh the downsides (Wang 2006; Wang *et al.* 2006). In principle, all measures that could further decrease water absorption and swelling of dried pulp fibers in water could help the dewatering of the fibers and the wet paper web in the papermaking, and in that way decrease papermaking energy consumption.

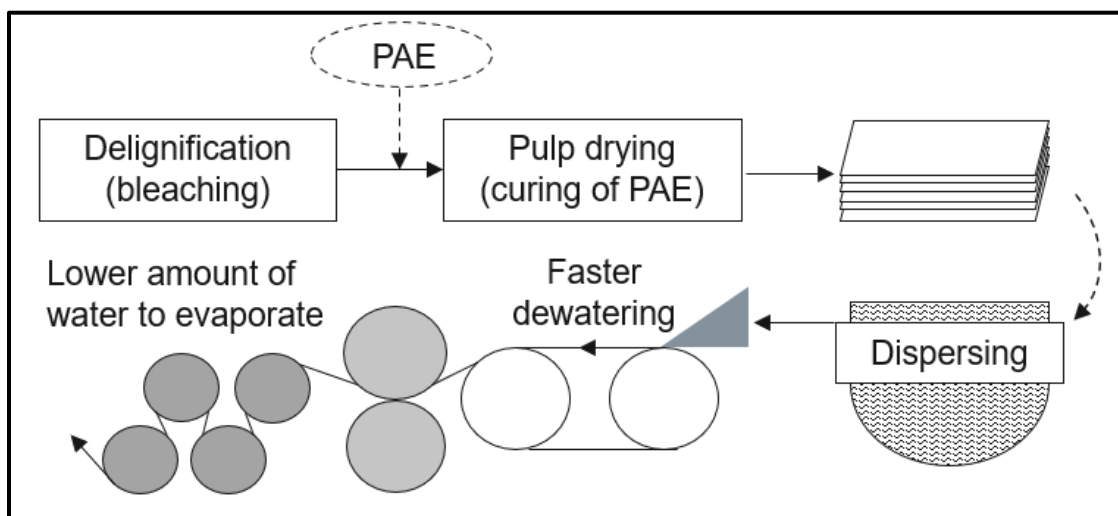
One of the known means to reduce kraft fibers' water absorption and swelling in water is fiber chemical crosslinking treatment. The method has some severe disadvantages though. In the treatment, chemical crosslinking agents, such as dialdehydes and polycarboxylic acids, are absorbed into the fiber walls, where they form water-resistant covalent bonds (crosslinks) between the adjacent cellulosic chains. The formed crosslinks prevent separation of the cellulose chains, and thus the swelling of the fiber walls, as they are wetted by water (Ward 1973; Caulfield 1994; Korpela and Tanaka 2015). Based on practical observation, Westervelt and Elston describe that dewatering of chemically crosslinked fibers can be notably rapid in the paper machine forming section. Without going into the used crosslinking chemicals and chemical dosages, Westervelt and Elston (1995) mention that in fourdrinier paper machines, crosslinked fibers' rapid drainage can cause "movement of the dry line all the way to the headbox". In a study by Korpela and Tanaka (2015), chemical crosslinking of birch kraft fibers with a methylated 1,3-dimethylol-4,5-dihydroxyethylene urea (mDMDHEU) crosslinking agent resulted in an increase of the fibers' Canadian Standard Freeness (CSF), from 525 mL to a maximum of 660 mL, and a decrease of WRV, from 1.60 to 1.20 g/g. The reported CSF and WRV measurements were taken after the PFI-refining (1000 rev.) of the pulps. In practice, a downside of the treatment is a relatively high required curing temperature, even when the appropriate catalysts are used. For example, in cotton-fabric crosslinking with customarily used mDMDHEU crosslinking agents, the typical curing temperature is around 140 to 150 °C and the curing time is 3 to 5 minutes. Another side effect of chemical crosslinking is loss of fiber-to-fiber bonding capability and embrittlement of the treated fibers (Westervelt and Elston 1995; Korpela and Tanaka 2015; Korpela and Orelma 2020). Probably due to the high required curing temperature and the side effects, the practical applications of chemically crosslinked kraft fibers have been mostly limited to specialty papers, such as filter papers and saturating kraft papers, and the manufacture of fluff pulps with improved porosity and wet resilience (Westervelt and Elston 1995; Lund and Breid 2014).

The aim of the present study was to clarify whether kraft fiber dewatering in paper machine forming and drying section could be enhanced by the polyamide-epichlorohydrin (PAE) treatment in the course of previous preparation of dry pulp. PAE is generally used in papermaking to increase the paper products' wet-strength. PAE is a cationic water-soluble polymer, which retains negatively charged papermaking fibers by ionic attraction. PAE is typically added to the papermaking furnish. The curing of PAE mostly takes place as the paper temperature is increased in the paper machine drying section (Espy 1995; Scott

1996). During the curing the PAE is believed to self-crosslink and to form water-resistant covalent bonds with cellulosic carboxyl groups. According to general understanding, PAE molecules increase the paper wet strength by repressing the fiber swelling and protect the fiber-to-fiber bonds from opening as the paper is wetted by water (Hägkvist *et al.* 1998; Ozaki *et al.* 2006; Siqueira 2012).

According to the hypothesis of the present study, the fiber-bound, self-crosslinked PAE polymer-network can restrain swelling and water absorption of the PAE-coated sulphate kraft fibers as they become wetted by water. This was tested by treating never-dried hardwood (HW) and softwood kraft fibers (SW) using “standard” PAE, “highly reactive” PAE, and “short chain” PAE. According to the PAE supplier’s definition, the “standard” PAE is a commonly used wet strength resin in the paper industry. In the “highly reactive” PAE, the number of reactive groups is somewhat higher than in the standard grade. This can have an increasing effect on the PAE-to-fiber covalent bonding and self-crosslinking degree of the cured PAE, and thus on the extensibility and flexibility of the PAE. The “short chain” PAE, on the other hand, is possibly capable of entering into smaller fiber wall pores than the longer chain “standard” and “highly reactive” PAEs. After the addition, the pulps were thickened by filtration, crumbled by hand, and dried. Finally, the PAE was cured in an elevated temperature at 80 °C. The cured dry pulps were dispersed in water, and the pulp WRV and °SR-value were measured.

In practice, the never-dried fibers could be treated with PAE in conjunction with pulp drying. In other words, unrefined kraft fibers could be treated with PAE before being prepared as a dry pulp product to be sold to paper mills. Since the adequate temperature for curing PAE is well below 100 °C, no separate curing stage is necessarily needed in pulp mills. This could be a significant advantage of the ideated PAE-treatment compared to the other kinds of chemical crosslinking. As in the case of chemically crosslinked pulps, dispersion of the PAE-treated dry pulps in water may necessitate special arrangements though. The industrial implementation of the ideated fiber PAE-treatment is illustrated in Fig. 1.



**Fig. 1.** Process layout of the ideated pulp treatment with PAE for improved paper dewatering and drying. Functioning of the idea was tested by laboratory scale experiments. The method is patent pending (Korpela and Asikainen 2023).

## EXPERIMENTAL

### Materials

Never-dried unbeaten bleached softwood and bleached hardwood sulphate pulps (SW and HW) were obtained from a Finnish pulp mill. The consistency of the SW and HW pulp was 10.2 wt.% and 8.9 wt.% as received, respectively. The used PAEs were Kymene 725EU, Kymene 920, and Soyad CA1130 obtained from Solenis Finland Oy (Tampere, Finland). All PAEs are technical grade products and were used as such in the laboratory trials. Ion-exchanged water was used for all dilutions. Because of the difficulty in the quantitative analysis of PAEs in the pulps, the effects of the PAE treatments on the dewatering and strength properties are considered as a function of the added amounts of the crosslinking agents in the pulp suspension.

### Application and Curing of PAE

The SW and HW pulps were diluted by water to 1.5 wt.% consistency. The pHs of the pulp dispersions were adjusted to neutral pH (6.5 to 7.5) using diluted HCL and NaOH for the adjustment. Diluted PAE (1.0 wt.%) was added into the pulp suspension, and pulp was mixed for one minute before filtering the suspension using a water jet vacuum pump connected to Büchner funnel with a filter cloth. The pulp cakes were crumbled by hand and dried overnight in a ventilated oven at 80 °C. To ensure complete curing of the PAE, all pulps were heated additionally in 80 °C for 2 h. The dry matter content of the HW and SW pulps were around 99 ±0.5% after the drying and heat treatment. The reference pulps were treated in a similar way but without a PAE addition. The PAE dosages (dry content) in the pulp suspension are shown in Table 1.

**Table 1.** Pulp PAE-treatments: PAE Dosages (dry content) in the Never-dried HW and SW Pulp Suspension on Dry Fiber Basis

Sample Code	Kymene 725EU	Sample Code	Kymene 920	Sample Code	Soyad 1130
HW ref.	-		-		-
HW A1	0.5%	HW B1	0.5%	HW C1	0.5%
HW A2	1.0%	HW B2	1.0%	HW C2	1.0%
HW A3	2.0%	HW B3	2.0%	HW C3	2.0%
SW ref.	-		-		-
SW A1	0.5%	SW B1	0.5%	SW C1	0.5%
SW A2	1.0%	SW B2	1.0%	SW C2	1.0%
SW A3	2.0%	SW B3	2.0%	SW C3	2.0%

HW ref., and SW ref. are control samples with no added PAE.

### Biodegradation Tests

The pulp samples' (Table 1) biodegradation rates were compared using a 48-h enzymatic hydrolysis test. The enzymatic hydrolysis test followed the method described by Leppänen *et al.* (2020). In the test, the pulp samples were hydrolyzed using a mixture of cellulase, mannanase, xylanase, and β-glucosidase enzymes for 48 h at 40 °C at 25 g/L solids content. An enzyme dose of 50 FPU/g was used. The degree of sample degradation (%) was calculated by comparing the amount of the liberated reducing sugars (g/L) to the initial amount of the sample (g/L). The sample size of the pulp was 100 mg. Two replicates of each sample were measured.

## Dispersion of the Pulps and Testing of Pulp and Handsheet Properties

For the WRV and °SR measurements, 30 g (dry) of each pulp were soaked overnight in 2 L of water (23 °C) followed by dispersion of the fibers by 60000 revs. in Lorenzen & Wetre wet-disintegrator (Type 981583, Stockholm, Sweden). The reported results are averages of two parallel measurements. The HW and SW pulp slurries were additionally dispersed for 2 min and 30 s using a Braun Multiquick 3 Hand Blender 450 W (De'Longhi Braun Household GmbH, Germany) for the handsheet making. Laboratory paper sheets were made using uncirculated ion-exchanged water following ISO 5269-1 (2005). The targeted handsheet grammage was 80 g/m<sup>2</sup> with an RH of 50%. To prevent splitting of the mechanically weak handsheets made of PAE-treated fibers, Teflon-coated drying plates were used in the handsheet making. The utilized test methods are shown in Table 2. Fiber fines (< 0.2 mm) contents were measured using an L&W FiberTester plus image analyzer (ABB Ab, Kista, Sweden) and confirmed using a Valmet FS5 image analyzer (Valmet Automation Inc, Kajaani, Finland). Microscopic examinations of the fibers were done using Nikon Model Eclipse Ci-L light microscope (Nikon Corporation, Tokyo, Japan) and Merlin FE-SEM scanning electron microscope (Carl Zeiss Microscopy GmbH, Oberkochen, Germany). The samples were sputter coated (EM ACE200, Leica, Germany) with 5 nm of Pd prior to SEM imaging.

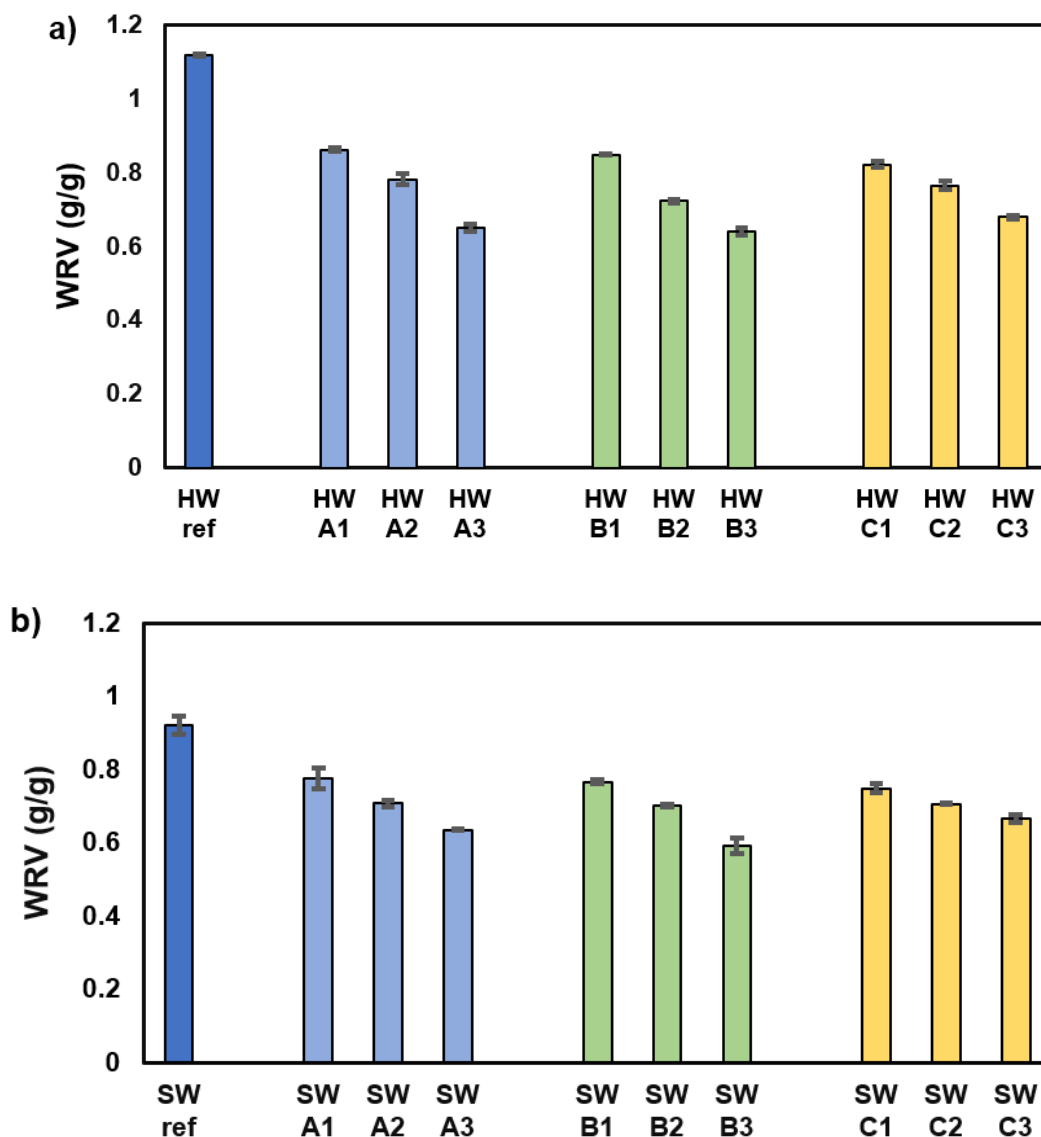
**Table 2.** Utilized Paper Test Methods

Water Retention Value (g/g)	ISO 23714:2007
Schopper-Riegler (°SR) value	EN ISO 5267-1:1999
Fines content (%)	ISO 16065-2:2014
Grammage (g/m <sup>2</sup> )	ISO 5270 (2012)
Bulk (kg/m <sup>3</sup> )	ISO 5270 (2012), ISO 536 (2019)
Tensile index (Nm/g)	ISO 5270 (2012)
Strain at break (%)	EN ISO 1924-3:2008

## RESULTS AND DISCUSSION

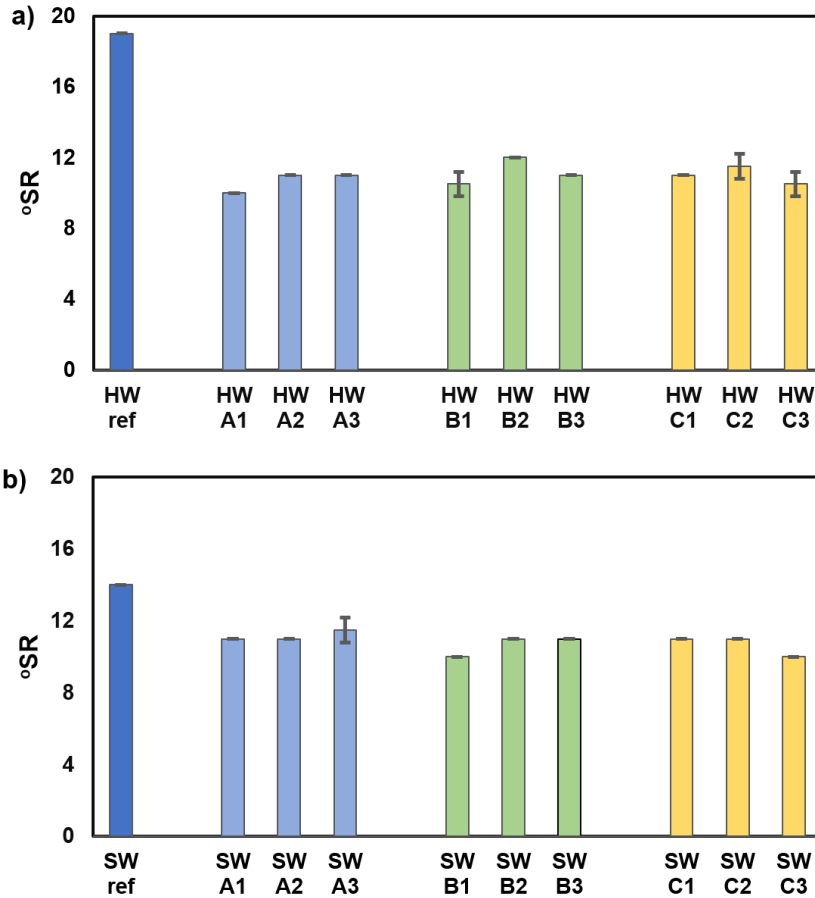
The results showed that the PAE treatment clearly lowered the WRV value of both HW and SW fibers, the relative effect being somewhat greater in the case of HW fibers (Fig. 2). The tested PAE-grades (“standard”, “highly reactive”, and “short chain”) had roughly the same effect on the fibers’ WRV values. According to the Shopper-Riegler measurement (°SR), PAE treatments also considerably affected the drainability of the HW and SW fibers (Fig. 3). A possible reason for this is the binding of inherent anionic fiber fines to the anionic fibers by the cationic PAE (Hubbe and Heitmann 2007; Liang *et al.* 2020). In the pulp suspension, the added PAE can thus act as a cationic retention agent and fixative. This explanation is supported by the considerable decrease of the number of fiber fines particles in the PAE-treated, re-dispersed pulp samples (Fig. 4). The decrease in the amount of fiber fines can also be seen in the photos taken with the Valmet FS5 (Fig. 5).

According to general understanding, fiber fines have a strong retarding effect on pulp drainage, and thus to the pulp °SR-value (Hubbe 2002; Hubbe and Heitmann 2007, Lindqvist *et al.* 2011). Unfortunately, analysis of fibers’ dimensional changes, such as fiber width, fiber wall thickness, and cross-sectional area, using the image analysis methods, are not necessarily reliable due to the absorption of the fiber fines on the fiber surfaces. Also, as can be seen in Fig. 5, pulps treated with PAE obviously did not disperse completely in water despite the enhanced pulp dispersion prior to the measurement.

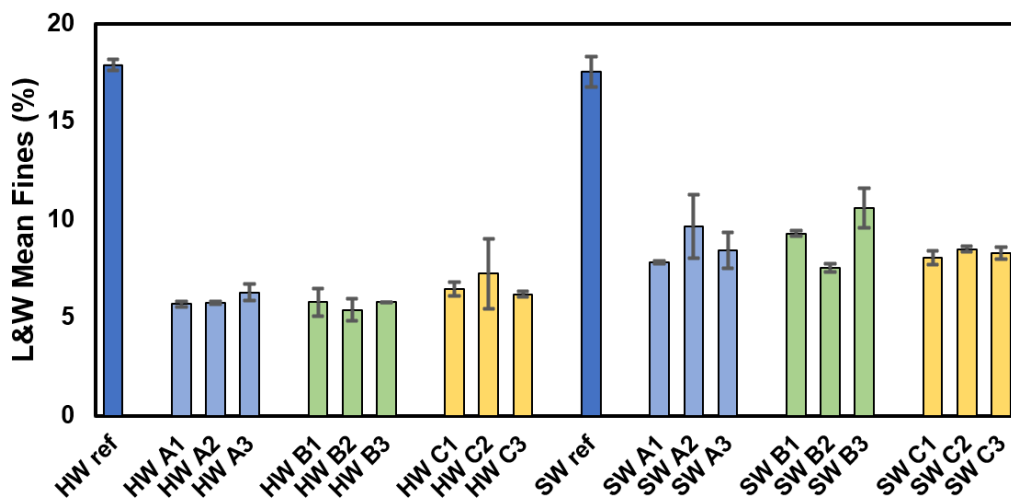


**Fig. 2.** The effect of a) HW and b) SW fibers' pretreatment by PAE on the fibers' water retention value (WRV). Letter "A" refers to "standard" grade PAE (Kymene 725EU), "B" to "highly reactive" PAE (Kymene 920), and "C" to "short chain" PAE (Soyad CA1130). Numbers 1, 2, and 3 refer to the added amount of PAE in the pulp suspension being 0,5 wt.%, 1,0 wt.%, and 2,0 wt.%, respectively.

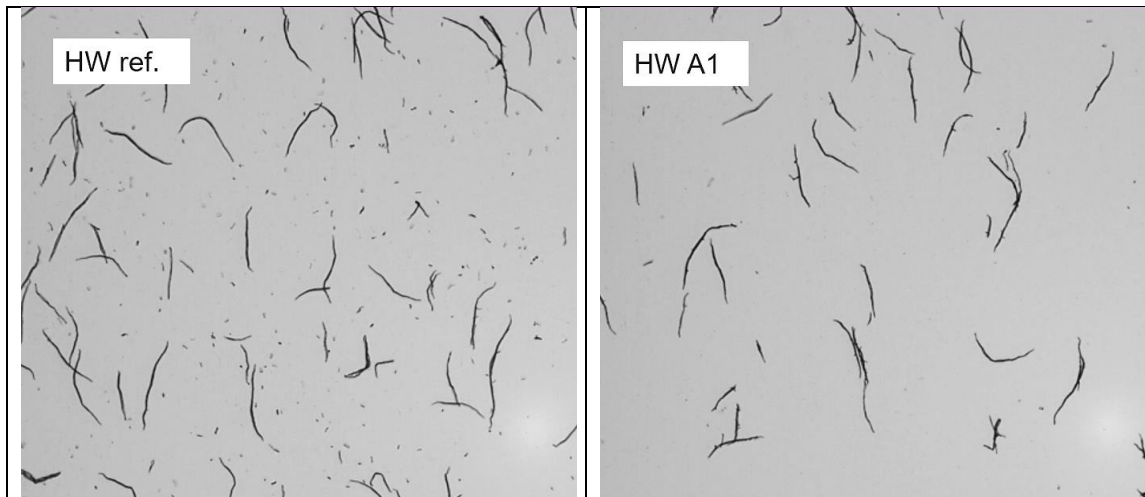
Based on the results in Figs. 2 and 3, PAE treatment increased the fiber suspensions' °SR-values and decreased the fibers' WRV. This further suggests that PAE treatment could improve the dewatering of wet paper web on the paper machine forming section and reduce the needed drying energy in the paper machine drying section, respectively. Unfortunately, the actual mechanism by which PAE lowers the fibers' WRV and °SR values remains somewhat uncertain. No clear difference between the reference fibers and the PAE-treated fibers was observed in light microscopy and SEM examinations either.



**Fig. 3.** The effect of a) HW and b) SW fibers pretreatment by PAE on the fibers Shopper-Riegler value (°SR). Letter “A” refers to standard grade PAE (Kymene 725EU), “B” to “highly reactive” PAE (Kymene 920), and “C” to “short chain” PAE (Soyad CA1130). Numbers 1, 2, and 3 refer to added amount of PAE in the pulp suspension being 0,5 wt.%, 1,0 wt.%, and 2,0 wt.% respectively.

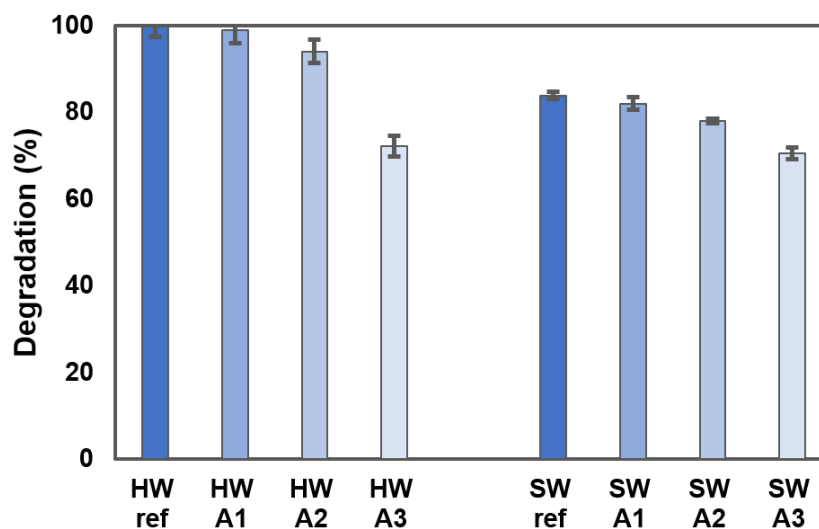


**Fig. 4.** The effect of a) HW and b) SW fibers’ pretreatment by PAE on the redispersed pulp mean fines% based on particles number distribution measured using L&W FiberTester Plus. Letter “A” refers to standard grade PAE (Kymene 725EU), “B” to “highly reactive” PAE (Kymene 920), and “C” to “short chain” PAE (Soyad CA1130). Numbers 1, 2, and 3 refer to the added amount of PAE in the pulp suspension being 0,5 wt.%, 1,0 wt.%, and 2,0 wt.%, respectively.



**Fig. 5.** Reference fibers (HW ref.) and PAE-treated fibers (HW 2A) pictured by Valmet FS5 Image analyzer. The amount of fine fiber particles is notably lower in the HW A2

Figure 6 shows the effect of the PAE treatments on the biodegradation rate of HW and SW fibers in the 48-h enzymatic hydrolysis test. According to the results, the increasing PAE-additions decreased the biodegradation rates consistently. However, the effect was not as strong as the effect of chemical cross-linking on the SW-handsheet biodegradation rate reported earlier by Korpela *et al.* (2023). According to the classification presented by Leppänen *et al.* (2020), the biodegradability of cellulosic materials, where the hydrolysis rate in the 48-hour hydrolysis test is in the range of 100 to 60%, 60 to 30%, and 30 to 0%, are “excellent”, “good”, and “poor”, respectively. Despite the decreased hydrolysis rate, all PAE-pretreated samples fell in the range of “excellent” biodegradability.



**Fig. 6.** The effect of PAE pretreatment on the biodegradation of HW and SW pulp in a 48-hour enzymatic hydrolysis test. Letter “A” refers to standard grade PAE (Kymene 725EU), Numbers 1, 2, and 3 refer to added amount of the PAE in the pulp suspension being 0,5 wt.%, 1,0 wt.%, and 2,0 wt.% respectively.



**Table 3.** Effect of PAE-Pretreatment on the Properties of HW and SW Pulps

	Tensile index (Nm/g)	Strain at break (%)	Bulk (cm <sup>3</sup> /g)
Unrefined bleached hardwood pulp (HW)			
HW ref	30.4 ± 1.9	4.00 ± 0.55	1.63 ± 0.04
HW A1 (PAE 0.5%)	6.7 ± 1.4	0.54 ± 0.09	2.31 ± 0.04
HW A2 (PAE 1.0%)	2.8 ± 0.4	0.38 ± 0.04	2.66 ± 0.05
Unrefined bleached softwood pulp (SW)			
SW ref	19.9 ± 1.2	4.69 ± 0.56	2.03 ± 0.02
SW A1 (PAE 0.5%)	6.3 ± 0.8	0.98 ± 0.16	2.62 ± 0.06
SW A2 (PAE 1.0%)	6.0 ± 0.8	0.98 ± 0.18	2.55 ± 0.04

Note: The PAE dosage (%) indicates the wt.% of an added sizing agent in never-dried pulp suspension on a dry fiber basis.

Based on the results, the research hypothesis was judged to be correct in that the performed PAE treatments reduced the WRV and °SR value of the water dispersed fibers. A side-effect of the treatment is a reduction in the fibers' bonding capability, resulting in decreased handsheet tensile strength and increased handsheet bulk (Table 3). Unfortunately, there is no complete certainty about the molecular level mechanism the PAE that makes the observed effects. Ouzaki *et al.* (2006), using confocal laser scanning microscope and special double staining method for detection of the PAE suggests that PAE increases paper wet strength by forming a fiber-bound, self-crosslinked coating on the fiber surfaces, which suppresses water-induced fiber swelling and protects fiber-to-fiber bonds from opening by water. In the present study, by using light microscopy and scanning electron microscopy, no clear and consistent differences between the reference fibers and the treated fibers' size or shape could be distinguished. No visible PAE-film on the fiber surfaces could be detected either. It is possible that the PAE acts not only on the fibers outer surface but in the fiber lumens and pores as well, thus preventing the collapsed lumens and pores from opening, as the PAE-treated fibers are redispersed in water. In his early study, Lindström (1980) suggests that the fiber walls swell mostly towards the fiber lumen by water, while the perimeter of the fibers remains the same. Also, it is conceivable that, at the molecular level, the PAE prevents the hydrogen-bonded cellulose “zippers”, described by Sjöstrand *et al.* (2023), from opening as the fibers are wetted by water. Such a “zipper lock” could prevent fiber swelling and fiber-to-fiber hydrogen bonds from opening as the paper is wetted. Unfortunately, it is very difficult to obtain direct information about the location and molecular level action of PAE on the fiber surfaces and inside the fibers.

Overall, the results of the present study suggest that the sulphate kraft fiber PAE treatment during a pulp drying operation could be used for improvement of the fibers' dewatering and bulking properties. Given the significant reduction in fiber-to-fiber bonding strength, PAE-treated fibers are likely unsuitable as the sole fiber component for producing ordinary printing and packaging papers. However, their good dewatering properties and high bulk can make them beneficial fiber component for enhancing these attributes in paper and board products where strong inter-fiber bonding is less critical. One such potential application for PAE-treated kraft fibers is in the middle layer of boxboard. Other potential applications of the specialty fibers include tissue papers, and absorbent materials. The focus of future research is to determine the suitability of the PAE-treated fibers for these applications and to investigate the response of the fibers to pulp refining and various dry and wet strength additives.

## CONCLUSIONS

1. Dewatering properties of unrefined sulphate kraft market pulps can be substantially improved by adding poly(amidoamine epichlorohydrin) (PAE) wet strength resin into fiber water suspension prior to pulp drying and subsequent curing of the PAE. Based on the water retention value (WRV) measurements, the PAE decreases fiber water absorption and fiber swelling. The fiber's decrease in degrees of Schopper Riegler ( $^{\circ}\text{SR}$ ) is likely due to fixation of fiber fines on the fiber surfaces and reduction of fines swelling by the PAE
2. The effects of the pulp PAE-treatment on the pulp dewatering and strength properties are like those of pulp chemical crosslinking treatment. The needed curing temperature of PAE is lower, making the treatment easier to implement in practice.
3. With enhanced dewatering properties and reduced fiber-to-fiber bonding capability, PAE-treated fibers could be well-suited as dewatering and bulking aid in boxboard middle layer as well as in tissue paper and absorbent materials.

## ACKNOWLEDGMENTS

This study was carried out in the Energy 1<sup>st</sup> fiber product forming research project funded by the European Regional Development Fund (ERDF) and participating companies. The ERDF and all involved companies are thanked for enabling the study. Maritta Räsänen (VTT), Päivi Sarja (VTT), Mervi Raatikainen (VTT), and Meiju Sinkkonen (VTT) are acknowledged for performing the fiber PAE treatments and testing of the pulp and handsheet properties. Ulla Salonen (VTT) and Hanna Christophliemk (VTT) are acknowledged for light microscopic and SEM examinations of the fibers. Elisa Spönla (VTT) and Riitta Alander (VTT) are acknowledged for performing of the enzymatic hydrolysis test and fungal resistance test.

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Article submitted: May 3, 2024; Peer review completed: May 26, 2024; Revised version received and accepted: June 7, 2024; Published: June 18, 2024.

DOI: 10.15376/biores.19.3.5227-5238