EFFECT OF SOFTWOOD KRAFT REFINING ON A MECHANICAL-CHEMICAL MIXTURE SHEET

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ABSTRACT

The main objective of this study was to maximize the fracture energy of a mechanical-chemical mixture sheet by softwood kraft refining. The second aim was to study the microscopic fracture behavior of paper during kraft refining.

According to the results, the kraft and mixture sheets are quite different. The fracture energy of mixture sheets decreases during softwood kraft refining, while that of pure kraft sheets increases. The damage analysis suggests an interpretation. First, the pull-out width of both mixture and pure kraft sheets decreases during refining. This indicates that the proportion of broken fibers increases. Second, in mixture sheets the fracture work per damage width stays almost constant during kraft refining, while in pure kraft sheets it is about three times larger at a high refining degree than for unrefined kraft. The study also showed that the fracture work of mixture sheets of TMP and unrefined kraft is larger than in pure TMP or unrefined kraft. However, at a higher refining degree, the fracture energy seems to follow roughly the linear rule of mixtures.

INTRODUCTION

It is well known how the refining affects the properties of pure chemical pulp, but mechanical-chemical pulp mixtures have been studied less. Especially the fracture toughness of mixture sheets is rarely discussed in the literature. Still, it is considered to be one of the most important runnability-related properties [1,2]. According to the linear elastic fracture mechanics fracture toughness (material property) can be calculated from fracture energy (G_C) and elastic modulus (E): $K_C = \sqrt{G_C E}$ [3]. The analysis of fracture energy was considered appropriate even though paper is not linearly elastic material. It should be noted that there exist little statistical data correlating fracture toughness to runnability and also the terms used are inconsistent (e.g. fracture toughness, fracture energy, fracture resistance). Earlier studies show that the fracture toughness of pure chemical pulp sheets has a similar maximum, though it occurs at a later stage of refining, as the conventional out-of-plane tear [1,4,5,6]. The different maximum is probably due to the different modes of testing (out-of-plane vs. in-plane) [1,7]. In mixture sheets the behavior of the fracture toughness is not clear, but the out-of-plane tear has had a small maximum during chemical pulp refining [8,9].

According to the linear rule of mixtures the mixture sheet properties would be linearly proportional to the component properties. In agreement with this rule, Kärenlampi et al. [10] have reported that the fracture toughness of a mixture of refined softwood kraft and pressure groundwood pulp is linearly proportional to the proportion of chemical pulp. On the other hand, Shallhorn [11] found the fracture resistance of a mixture of unrefined softwood kraft and groundwood pulp to be non-linear: the fracture resistance was higher than predicted from the linear rule of mixtures. Also other sheet properties have been found to be non-linear in relation to mixture proportions [8,12,13]. According to Parsons [8] and Mohlin et al. [12], chemical pulp behaves in mixtures as if it would have been refined less than it actually is: the increase in tensile strength was smaller and in out-of-plane tear higher than expected from the linear additivity relationship.

The original working hypothesis was based on the above observations. It was assumed that refining the chemical pulp component could increase the fracture energy of paper made from a mixture of mechanical and chemical pulps. However, initial results [14] indicated that the fracture energy of mixture sheets did not increase through softwood kraft refining.

In the present study, the working hypothesis was further checked with two types of reinforcement pulp. In addition to TCF-bleached kraft, also unbleached softwood kraft was used to eliminate the uncertainty related to fiber damage that may be caused in pulp bleaching. Furthermore, the microscopic damage in paper during kraft refining was studied to determine the mechanisms affecting fracture energy. A novel analysis method was used for this purpose: the width of the damage zone (or the fracture process zone) and the pull-out zone (fiber ends that stick out from the crack line) were measured [15]. Relatively high proportions of chemical pulp were used (40% and 50%). This corresponds with the typical range of LWC base paper.

EXPERIMENTAL

Raw material

TCF-bleached softwood kraft (dried pulp, 50% Scandinavian spruce and 50% Scandinavian pine) was obtained from a Finnish pulp mill. The length-weighted average fiber length (Kajaani FS-100) of unrefined pulp was 2.17 mm and the coarseness 0.170 mg/m. Unbleached softwood kraft (never dried, 45% Scandinavian spruce and 55% Scandinavian pine) was obtained from another Finnish pulp mill. The average fiber length of unrefined pulp was 2.21 mm (length-weighted) and the coarseness 0.180 mg/m. TMP (Scandinavian spruce) was obtained from a Finnish paper mill at a consistency of 8% and stored in freezer to await experiments. The CSF value was 40 (SCAN-C 21:65) and the length-weighted average fiber length was 1.59 mm. The coarseness of the R30 fraction was 0.222 mg/m.

Refining

Chemical pulp (4 % consistency) was refined in an Escher-Wyss (EW) conical laboratory refiner. With the EW refiner it is possible to control the amount of refining energy and refining intensity separately. The intensity was calculated according to the Specific Edge Load theory (SEL). Certain practical aspects of this theory have been discussed by Baker [16]. Softwood kraft pulps were refined both at low and high intensity levels (1.0 J/m and 4.0 J/m) and the refining energy was varied in the range 0 - 200 kWh/t.

Sample material

Handsheets were prepared from pure chemical pulp, pure mechanical pulp and from mixtures of the two. Different mixture ratios were used: 40% TCF - 60% TMP and 50% unbleached kraft - 50% TMP. Sheets containing mechanical pulp were prepared with a laboratory handsheet mold equipped with a white water circulation system [17].

Mechanical testing

Fiber strength was estimated by measuring the wet zero-span tensile strength (Tappi T 273 pm-95). Ordinary sheet properties were measured in accordance with the standards SCAN-P 7:75 (density), SCAN-P 11:73 (out-of-plane tear, Elmendorf, four parallel measurements), SCAN-P 38:80 (tensile strength, breaking strain and tensile stiffness).

The fracture energy was measured using two tests (four parallel measurements in both): the in-plane tear test [4,18,19] and the J-integral [20]. The in-plane tear test was done with an ordinary tensile testing machine (MTS 400/M). The initial span length was 45 mm and specimen width 70 mm. The tight edge of the specimen had a 15-mm notch in the middle. Thus, the ligament length was 55 mm. The displacement rate was 20 mm/min and the tearing angle 12°. Details of the test are given in another report [19]. The J-integral was measured with a Lorentzen & Wettre Fracture Toughness tester (SCAN-P 77:95). It is difficult to judge which of the two fracture methods that describes better the runnability of a dry paper web. The principal difference between the tests is that the in-plane tear measures the energy consumed at the fracture process zone when crack propagates through the specimen, while the J-integral characterizes energy dissipation at the onset of crack propagation [19,21]. In earlier studies [19,21], the in-plane tear work was observed to be larger than fracture energy measured by the J-integral test.

Damage analysis

The in-plane tear test was also done for silicone-impregnated samples in order to measure the damage width and the pull-out width [15]. Silicone enhances the contrast of newly revealed fiber surfaces enough to allow the interfiber bond openings and fiber breakage to be detected [22]. Silicone impregnation has only a minor effect on the interfiber debonding process. After tearing, the samples were scanned with an ordinary desktop scanner (Mustek). The images were analyzed using an appropriate program. Two parameters are measured: Damage width characterizes the extent of fiber debonding from the crack line. Pull-out width characterizes the distance of the sticking fiber ends from the crack. For further details see [15].

RESULTS

In addition to the graphs in this chapter, the measured results and the corresponding standard deviations are shown in the table of Appendix 1.

Tensile strength and fracture energy

Refining of the softwood kraft component increased the tensile index of both pure kraft sheets and mixture sheets, as shown in **Figure 1**. The increase was smaller in the mixture series than in the pure kraft series, probably because the tensile index of pure TMP was higher than that of unrefined kraft. The trend lines of TCF and unbleached kraft were qualitatively similar both in the pure kraft and the mixture series. The pure unbleached kraft was stronger than TCF, but the mixture series are not comparable because of different TMP proportions.

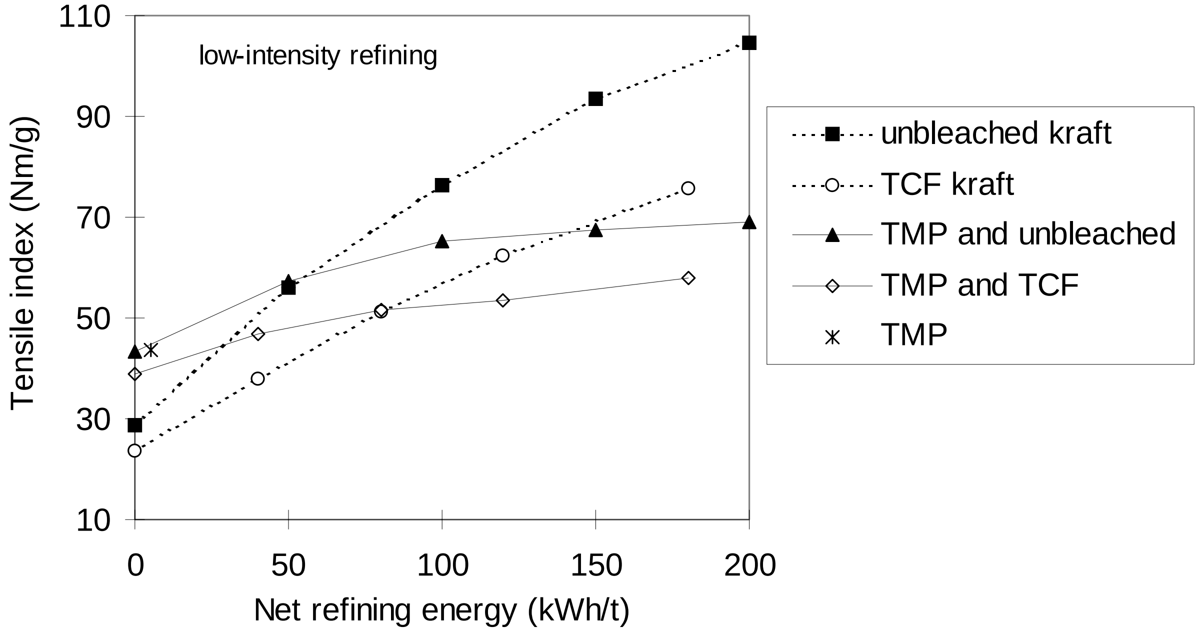

Figure 1. Tensile index of the kraft and the mixture series vs. refining energy of softwood kraft. Note the mixture proportions: 40% TCF - 60% TMP and 50% unbleached kraft - 50% TMP.

The out-of-plane tear work of both kraft and mixture series decreased with kraft refining, as shown in **Figure 2**. In earlier studies by authors' group [14,23] the trend line of the mixture sheets had no maximum, not even with very low refining energy, though a small maximum was observed for pure TCF kraft. However, Parsons [8] has reported a maximum for the out-of-plane tear work in mixture sheets. He used SGW of lower tensile index than that of the TMP used in this study.

The in-plane tear work of the softwood kraft series developed differently from that of the mixture series during kraft refining, as shown in **Figure 3**. Pure kraft series reached a maximum at a rather high refining degree (75 - 150 kWh/t), while the mixture series did not show a maximum. The in-plane tear work decreased slowly during kraft refining. The trend lines did not change in high-intensity refining (see Appendix 1).

Figure 3 also shows that the fracture energy of the unrefined unbleached kraft - TMP mixture is larger than the corresponding component values. The linear rule of mixtures seems applicable only at a high refining degree, above about 75 kWh/t.

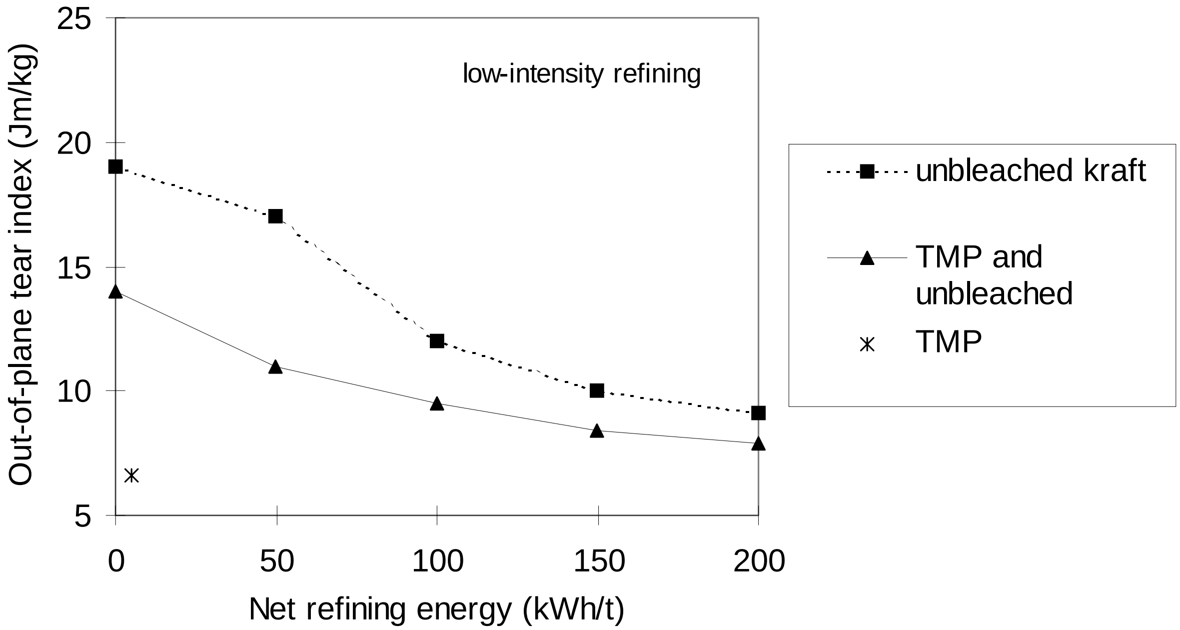

Figure 2. Out-of-plane tear index (Elmendorf) of the unbleached kraft series and the mixture series vs. refining energy of softwood kraft.

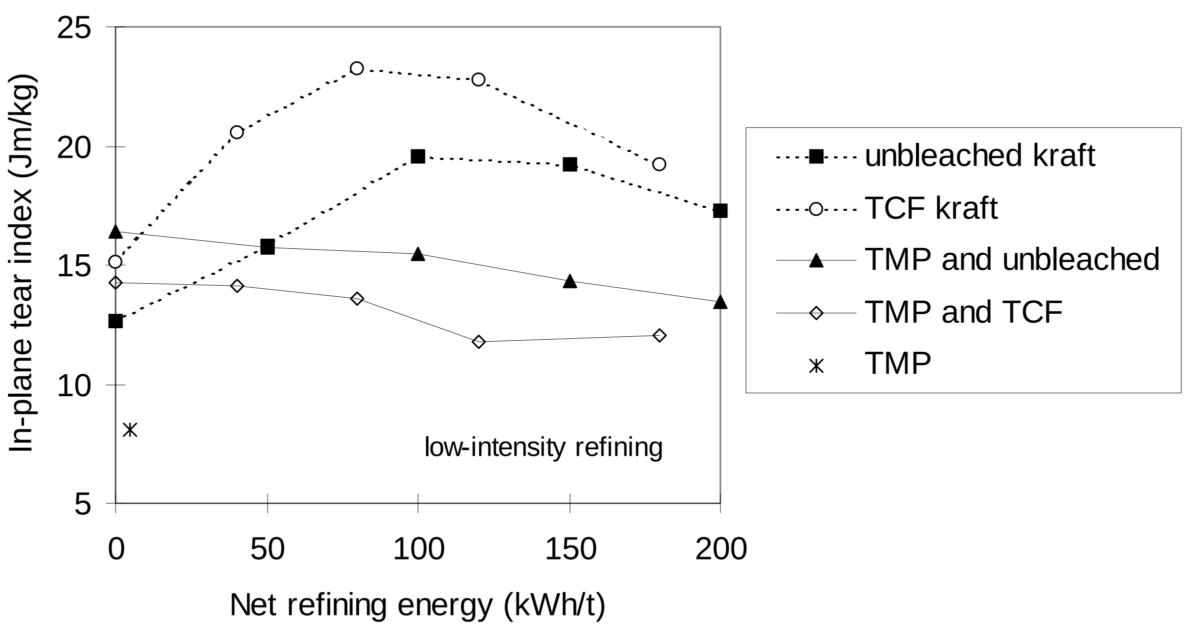

Figure 3. In-plane tear index of the kraft and the mixture series vs. refining energy of softwood kraft.

The trend lines of the in-plane tear work differ little from L&W J-integral results during refining of the unbleached softwood kraft, as shown in **Figure 4**. The J-integral gives lower values than the in-plane tear, which is in agreement with earlier results [19,21]. Both measurements show a clear increase in the fracture work in the pure kraft series but only small changes in the mixture series during refining. The J-integral maximum of the kraft series seems to correspond to a slightly lower refining degree. Also the J-integral of the mixture series showed some deviation. However it should be noted that the deviation of the measurements is large due to limited amount of sample material. Thus it is difficult to say if the qualitative differences are true.

Microscopic damage

The damage width of the silicone-impregnated samples decreased during kraft refining, both with the pure kraft and the mixture series, as shown in **Figure 5**. However, the change was much smaller in the mixture series than in the pure kraft series. This indicates that the interfiber bonding degree increased in the pure kraft sheets during refining, whereas the refining affected only slightly the bonding degree of the mixture sheets.

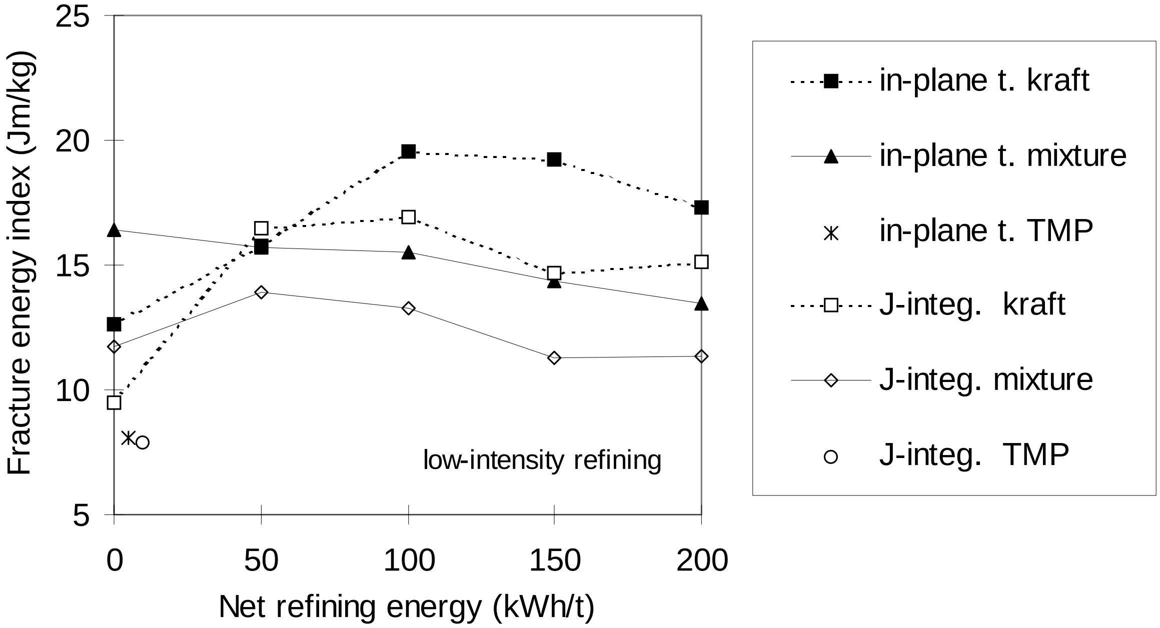

Figure 4. In-plane tear and L&W J-integral index of the kraft and the mixture series vs. refining energy of unbleached softwood kraft.

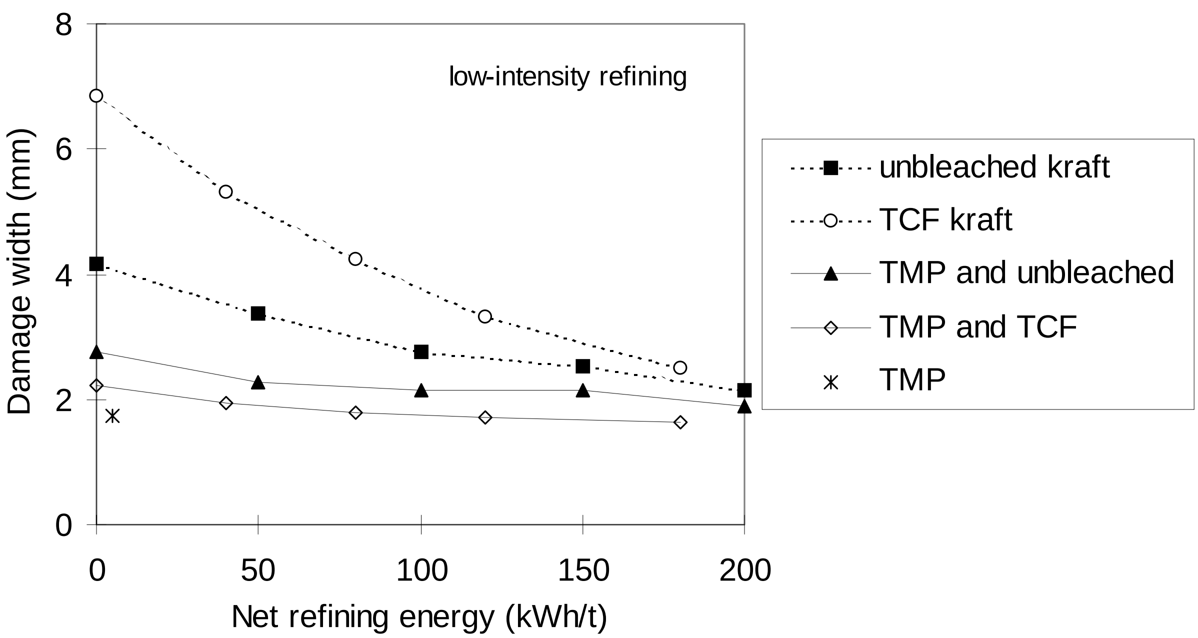

Figure 5. Damage width of the kraft and the mixture series vs. refining energy of softwood kraft.

Figure 6 shows the change in pull-out widths with increased refining. The trend lines indicate that the proportion of broken fibers increases (compared to fibers being pulled-out as a whole) both in the pure kraft and the mixture series right from the beginning of kraft refining. However, the in-plane tear work and the J-integral of the pure kraft series increased clearly at a low refining degree (Fig. 4). This shows that the fracture energy consumption related to interfiber bond ruptures increases more than energy is decreased by the additional fiber breakage. At a certain refining degree a maximum is reached and the subsequent fracture work decreases.

Figure 7 shows that the fracture energy per damage width of pure kraft series increases during kraft refining. The trend line seems to level off at about 100 kWh/t, which is close to the maximum of the in-plane tear work (Figure 3). However, the fracture energy per damage width of the mixture series is almost unaffected by kraft refining. It is at a high level already with unbeaten kraft.

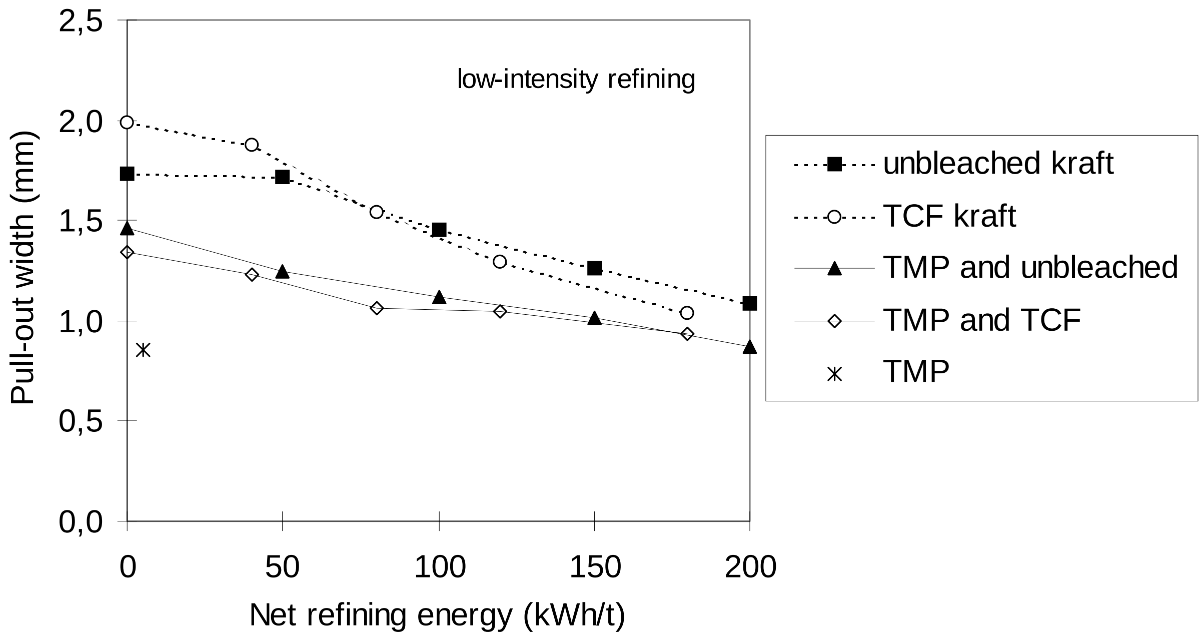

Figure 6. Pull-out width of the kraft and the mixture series vs. refining energy of softwood kraft.

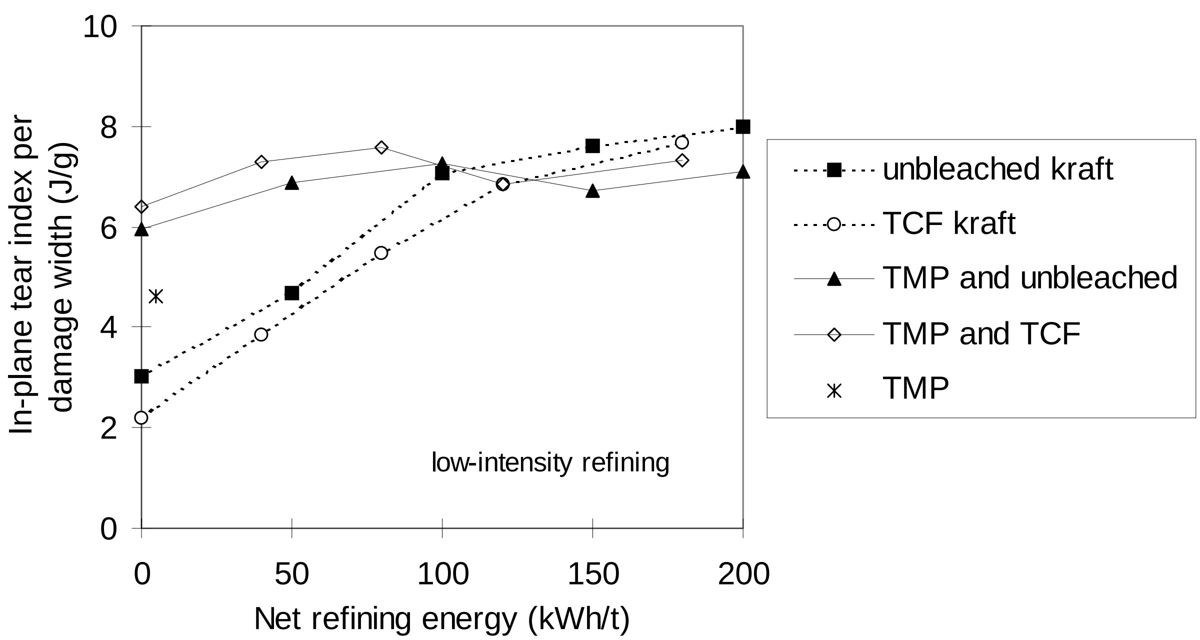

Figure 7. Energy density index (in-plane tear index/damage zone width) of the kraft and the mixture series vs. refining energy of softwood kraft.

DISCUSSION

In the working hypothesis it was assumed that kraft refining could increase the fracture energy of a TMP - kraft mixture. However, the results did not indicate this. The fracture energy of the mixture series decreased or remained constant during kraft refining. The tensile index and the tensile stiffness indicated that the interfiber bonding of the mixture sheets of TMP and unrefined kraft is stronger than that of the unrefined kraft. The interfiber bonding may be strong enough that the ratio of fiber breakage governs the fracture process against fiber debonding already with unbeaten kraft.

The experiments also showed that the fracture energy of the mixture sheets did not follow the linear rule of mixtures. The fracture energy of the TMP - unrefined kraft mixture was higher than the energy of either of the components. It is plausible that a suitable combination of long fibers (kraft) and high bonding capacity fines (TMP) leads to non-linear mixture behavior. However, at a higher refining degree, above about 75 kWh/t, the rule seemed valid.

The results of the damage analysis show that the fiber breakage increases (the pull-out width decreases), both in the pure kraft and the mixture series during kraft refining. However, fiber breakage does not alone explain the difference between the fracture energy behavior of the kraft and the mixture series. Though the pull-out width of kraft decreased from the beginning of refining, the fracture energy reached a maximum during refining. The fracture energy per damage width of the kraft series increased clearly with kraft refining, while that of the mixture series was almost constant. Apparently in the mixture sheets, kraft refining does not increase the energy needed to open interfiber bonds. However, at the same time, fiber breakage increases, causing a slight decrease in the fracture energy. In summary: In the mixture series the in-plane tear work per damage width remains constant even though the damaged area becomes smaller.

Based on this and on other studies [10,21,24,25] the fiber properties (e.g. length, strength) of the chemical reinforcement pulp can be assumed to be more important for fracture energy in a highly refined TMP- kraft mixture than the refining of chemical pulp.

Future studies

The TMP used in this study was highly refined, which is typical in LWC base paper production. If the interfiber bonding of sheets were decreased e.g. by fines removal or filler addition, the effect of kraft refining could be different. Further tests are being planned to examine this assumption.

CONCLUSIONS

The interfiber bonding of mixture sheets (TMP - kraft) can be increased by refining the kraft component. This is seen both from the bonding-dependent tensile properties and from the damage analysis. Refining could also increase the fracture energy of pure softwood kraft sheets. However, the fracture energy of mixture sheets did not increase during kraft refining. It seems that kraft refining does not produce a sufficient increase in the energy related to interfiber bond openings in the mixture sheets, though refining causes increased fiber breakage.

The fracture behavior of mixture sheets did not always follow the linear rule of mixtures. In some cases the fracture energy was higher than that of either of the components.

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APPENDIX 1. Results table

net refining energy	fiber length	CSF	gramm.	Density		Tensile index		Stretch		Tensile stiffness i.		Elmendorf-tear index		Wet zero span i.		In-plane tear index		L&W J-integral		Pull-out width		Damage width	
[kWh/t]	[mm]		[g/m²]	[kg/m³]	Stdev	[Nm/g]	Stdev	[%]	Stdev	[Nm/g]	Stdev	[Nm²/kg]	Stdev	[Nm/g]	Stdev	[Jm/kg]	Stdev	[Jm/kg]	Stdev	[mm]	Stdev	[mm]	Stdev
Unbleached softwood kraft, low refining intensity (1,0 J/m)																							
0	2.22	705	64.9	580	5	28.6	2.2	2.4	0.3	4050	210	19.0	2.3	120	6	12.6	0.5	9.5	0.9	1.74	0.14	4.16	0.26
50	2.31	630	65.2	644	7	56.0	2.7	3.2	0.2	6000	126	17.0	1.5	126	7	15.8	0.8	16.5	2.1	1.71	0.11	3.37	0.09
100	2.30	510	66.2	715	7	76.3	3.9	3.2	0.2	7220	322	12.0	0.6	123	7	19.5	0.5	17.0	3.1	1.45	0.06	2.76	0.15
150	2.29	355	64.8	760	3	93.3	3.0	3.3	0.3	8250	182	10.0	0.6	131	6	19.2	0.2	14.7	0.4	1.26	0.09	2.52	0.05
200	2.25	206	64.9	791	3	104.7	5.3	3.5	0.2	8850	238	9.1	0.4	123	3	17.3	0.7	15.1	0.6	1.09	0.05	2.16	0.09
Unbleached softwood kraft, high refining intensity (4,0 J/m)																							
0	2.20	715	69.4	571	6	26.2	1.6	2.5	0.3	3630	98	17.0	2.5	107	6	11.3	0.8	-	-	1.80	0.16	4.00	0.11
50	2.20	650	65.5	663	7	55.6	2.1	3.0	0.2	6290	140	17.0	1.0	121	9	17.6	0.6	-	-	1.71	0.11	3.23	0.21
100	2.24	570	64.3	679	6	64.8	1.8	3.2	0.3	6870	196	14.0	0.4	130	4	18.7	0.5	-	-	1.49	0.04	2.92	0.21
150	2.16	450	65.2	742	5	78.8	3.8	3.6	0.2	7680	280	11.0	0.1	117	6	18.6	0.4	-	-	1.34	0.02	2.44	0.09
200	2.10	305	65.8	775	5	85.8	2.9	3.7	0.1	8100	294	9.0	0.3	115	4	16.9	0.5	-	-	1.07	0.04	2.02	0.02
TCF softwood kraft, low refining intensity (1,0 J/m)																							
0	2.17	665	63.0	572	5	23.6	0.8	3.2	0.2	2998	77	-	-	-	-	15.1	0.8	-	-	1.99	0.06	6.86	0.69
40	2.09	620	65.0	621	5	37.9	1.3	3.7	0.2	3958	96	-	-	-	-	20.5	0.4	-	-	1.87	0.04	5.32	0.01
80	2.02	510	63.7	656	5	51.3	2.5	3.8	0.3	5367	160	-	-	-	-	23.3	1.4	-	-	1.55	0.00	4.24	0.10
120	1.95	365	63.1	708	7	62.5	3.8	3.8	0.3	6190	218	-	-	-	-	22.8	0.9	-	-	1.29	0.03	3.33	0.04
180	-	180	64.9	759	6	75.8	2.1	3.9	0.2	7111	252	-	-	-	-	19.2	1.0	-	-	1.04	0.03	2.51	0.19
TMP																							
	1.59	47	69.5	474	1.399	43.7	3.6	2.1	0.3	4210	280	6.6	0.3	74	3	8.1	0.4			0.86	0.04	1.75	0.24
Mixture sheets, 50 % TMP + 50 % unbleached kraft, low refining intensity																							
0	-	-	66.5	553	7	43.5	2.4	2.9	0.5	4672	115	14.0	1.4	-	-	16.4	0.2	11.8	1.8	1.46	0.04	2.75	0.09
50	-	-	66.7	583	3	57.3	2.6	3.2	0.2	5354	121	11.0	0.8	-	-	15.7	0.7	13.9	2.1	1.25	0.04	2.29	0.12
100	-	-	67.5	620	4	65.3	8.4	3.2	0.7	5992	177	9.5	0.9	-	-	15.5	0.5	13.3	0.9	1.12	0.07	2.14	0.13
150	-	-	67.8	628	4	67.6	2.3	3.2	0.2	5935	200	8.4	0.2	-	-	14.4	0.2	11.3	0.1	1.02	0.06	2.14	0.18
200	-	-	65.7	653	4	69.0	3.0	3.1	0.3	6105	106	7.9	0.3	-	-	13.5	0.5	11.3	0.3	0.87	0.02	1.89	0.06
Mixture sheets, 50 % TMP + 50 % unbleached kraft, high refining intensity																							
0	-	-	68.7	550	3	40.8	2.1	3.0	0.4	4358	173	15.0	0.4	-	-	16.1	0.4	-	-	1.51	0.04	2.84	0.11
50	-	-	68.1	580	6	52.7	2.2	3.3	0.2	5036	355	12.0	0.3	-	-	14.9	0.7	-	-	1.27	0.07	2.36	0.11
100	-	-	68.0	602	5	55.8	3.2	3.1	0.2	5347	127	10.0	0.0	-	-	15.5	0.9	-	-	1.16	0.02	2.23	0.06
150	-	-	67.8	617	3	59.1	2.7	3.4	0.1	5398	183	9.1	0.2	-	-	14.6	0.5	-	-	1.03	0.02	2.10	0.08
200	-	-	69.0	640	7	63.3	2.2	3.3	0.2	5728	146	8.5	0.5	-	-	13.9	0.7	-	-	0.94	0.06	1.92	0.09
Mixture sheets, 60 % TMP + 40 % TCF kraft, high refining intensity																							
0	-	-	68.4	547	3	38.8	1.5	2.8	0.3	4012	80	-	-	-	-	14.3	0.1	-	-	1.34	0.07	2.22	0.09
40	-	-	67.9	571	4	46.8	2.5	3.2	0.3	4478	180	-	-	-	-	14.1	0.1	-	-	1.23	0.06	1.94	0.01
80	-	-	68.9	590	3	51.6	2.4	3.1	0.3	4818	324	-	-	-	-	13.6	0.8	-	-	1.06	0.03	1.80	0.00
120	-	-	68.2	599	5	53.6	2.1	2.9	0.2	5324	289	-	-	-	-	11.8	0.1	-	-	1.05	0.04	1.71	0.06
180	-	-	69.3	633	5	57.9	2.5	3.1	0.2	5522	148	-	-	-	-	12.0	0.1	-	-	0.93	0.00	1.64	0.10