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Polymers

Fiber length and bonding effects on tensile strength and toughness of kraft paper

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ABSTRACT

Fiber length and fiber-to-fiber bonding effects on tensile strength and fracture toughness of kraft paper have experimentally been investigated. Laboratory sheets were made from kraft pulp, each with a distinct set of fiber lengths. Additionally, the fiber–fiber bond strength was improved by carboxymethyl (CMC) grafting. The tensile strength and work of fracture toughness results were compared to predictions from a shear-lag model which considers the fiber–fiber bond shear strength, the fiber tensile strength and fiber pull-out work. The tensile strength and fracture work for papers with weak fiber–fiber bonds increased with fiber length consistent with the shear-lag model. CMC-treated fibers provided strong fiber–fiber bonds. Papers made from such fibers displayed high strength and work of fracture independent of fiber length which indicates that the failure process is governed by fiber failures rather than bond failures. The fracture toughness, expressed as the critical value of the J-integral, increased strongly with fiber length for both untreated and CMC-treated papers. The results show that long fibers and CMC addition are extremely beneficial for improving the fracture toughness.

Introduction

Early models for the prediction of the tensile strength of paper were developed by, e.g., Kallmes et al. [9, 10] and Page [19]. Such models provide insight on how material and processing parameters influence the tensile strength but suffer from several simplifying assumptions. Other more physically based approaches, such as the shear-lag concept, introduced by Cox [3], where tensile stress in a fiber is gradually introduced by shear stresses at fiber–fiber bonds, have been applied to tensile strength analysis of unidirectional short-fiber composites by Kelly and Tyson [12]. There are several structural similarities between a short-fiber composite and paper. The fibers in a short-fiber composite are bonded to a matrix, while the short fibers in paper are bonded to each other. For a tensile loaded composite consisting of short fibers that are weakly bonded to the matrix, the fibers will pull out from the matrix rather than fracture. Papers where the fibers are held together by weak bonds will similarly fail by shear failure of the

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bonds if the fibers are strong. These similarities were recognized by Shallhorn and Karnis [29] and de Ruvo et al. [4] and formally incorporated in an analysis of the tensile strength of paper by Carlsson and Lindstrom [2]. Predictions from this model were in good agreement with previously published experimental results by Ingmanson and Thode [8] and Watson and Dadswell [37].

A central concept in the shear-lag theory is the critical fiber length, l_c, i.e., the minimum fiber length required to transition of the failure process from fiber pull-out to fiber fracture which benefits strength of the material if the fibers are strong. Papers with strong bonds have small values of l_c, which implies that long fibers are not required to obtain strong papers. This model predicts that the strength of paper increases with fiber length, with a rate depending on the bond strength; see Carlsson and Lindstrom [2].

Chemical additives (dry strength agents) are used in papermaking to increase bond strength and improve printability, etc., see, e.g., [5, 16, 17]. Several chemicals improve the shear strength of fiber–fiber bonds as well as increasing the relative bonded area (RBA). Surface carboxymethylation (CMC) is a method developed by Laine et al. [14, 15] to improve fiber–fiber bond strength. CMC addition also helps to disperse the fibers [35, 36]. Fiber flocculation problems are known to deteriorate with increasing fiber length, but in the current experiments with CMC-grafted fibers the formation was maintained.

In this paper, a systematic study on the effects of fiber length and fiber-to-fiber bonding on the tensile strength and fracture toughness of paper is presented. We prepared a set of papers from fibers of specific lengths and varied the fiber–fiber bond strength by addition of CMC. The papers were tested under in-plane tension to examine the influences of fiber length and bonding on stiffness and strength. In addition, in-plane fracture testing was conducted.

Fracture toughness of paper

While the mechanism of tensile failure of paper is fairly well understood (see above), the strength of paper containing a sharp crack, as shown in Fig. 1, is less understood in terms of fiber and bond properties of paper. Extension of a crack in a paper network is obstructed by a tortuous path due to the heterogeneous structure of the material. Crack propagation

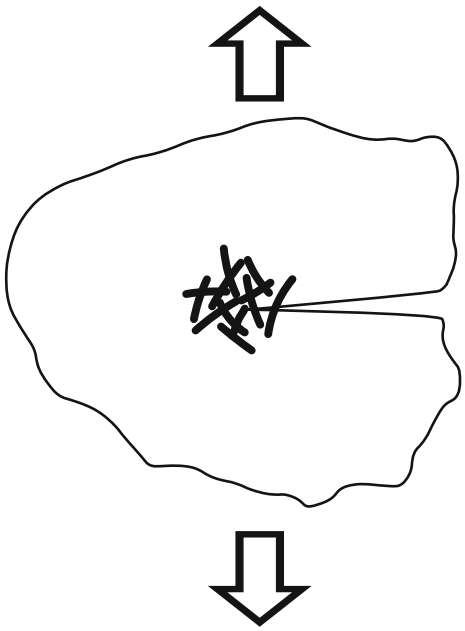

Figure 1 Crack propagation in paper.

involves fiber pull-out and fiber fractures in a narrow zone several mm in diameter close to the crack tip, Niskanen et al. [18]. Plastic yielding of the material near the crack tip is also occurring [38]. Such mechanisms require nonlinear fracture mechanics models. The J-integral proposed by Rice [23] has emerged as a method to incorporate nonlinear crack tip elastic–plastic stress and strain fields in the fracture mechanics analysis. The resistance of the material to crack propagation (J_c) is called fracture toughness. A major reason for the popularity of the J-integral method is that it is readily calculated from the global load–displacement record of a cracked specimen. This method was for applied to fracture of paper by, e.g., Uesaka [34], Steadman and Fellers [31], Westerlind et al. [39], and Wellmar et al. [38].

With respect to fracture toughness as measured by J_c, mechanisms such as damage formation, e.g., fiber pull-out, and plastic yielding contribute to nonlinear behavior of the material near the crack tip and are known to substantially increase the fracture toughness. In fact, for ductile materials, the plastic strain work accompanying crack extension largely dominates the fracture resistance [6].

The influences of fiber length and fiber–fiber bond strength on fracture toughness measured by the J-integral require further study. Fracture toughness, characterized by the work of fracture of an uncracked short-fiber composite, was approached by an

extension of the shear-lag model by Kelly [13]. The work of fracture is a measure of the energy required to fully separate an uncracked specimen, which is different from the fracture toughness, J_c, which measures the resistance to propagate a sharp crack. Kelly [13] assumed that the work done in separating a specimen in two parts is consumed by frictional sliding at the fiber/matrix interface (after bond breakage). It was assumed that breakage of fibers consumed negligible work compared to the pull-out work. Shallhorn and Karnis [30] calculated "tear strength" (fracture work) of paper based on similar assumptions. Analysis of the influence of fiber length predicts that the work of fracture increases with fiber length from zero at zero fiber length until a sharp maximum is reached when the fiber length equals the critical length.

Materials and test procedures

Materials

A stem of a 70–80-year-old spruce tree was chopped about two meters above the ground, from which a 1-m-long log was cut. The round log was then trimmed to achieve a 20 × 20 (cm) × 1 m piece which was then cut into four axially oriented bars of length 1 m with a 5 × 5 (cm) cross section. To achieve desired fiber lengths, chips were cut transverse to the length axis of the bars (perpendicular to the fiber direction). The length of spruce fibers ranges from 1.7 to 3.7 mm with an average of 2.9 mm [7]. Hence, in order to extract intact fibers the chip thickness should exceed 3.7 mm. Chips of thicknesses 1, 2, 3, 4 and 5 mm were cut from the bars. In addition, a set of chips with different thicknesses (2–8 mm) was prepared. This reference consists of fibers with lengths between 1 and 5 mm and should be representative for ordinary paper. Each set of chips was laboratory-cooked by conventional kraft cooking (the charge was 19% effective alkali and 35% sulfidity, and the H-factor was 1900) followed by a DEDED ECF bleaching sequence. For details on pulp preparation, see Biermann [1]. Kappa number and yield were determined for the various fiber length fractions. The Kappa number was determined as the average from two samples. The yield was based on the entire batch. The Kappa number varied slightly, from 23.7 to 27.5, and the yield varied from 49.7 to 52.0%. Hence, the consistent cooking and bleaching operations of the chips produced fibers with uniform chemical composition. Brightness and viscosity of the pulps were determined based on average from two samples. ISO brightness varied in a small range, 89.3–90.6%, and viscosity varied from 1080 to 1150 ml/g. The fiber length and the shape factor were measured with STFI FiberMaster [11]. The shape factor is defined as the ratio between end-to-end distance, d, of a fiber and the actual fiber length, l_f. This ratio is a number between 0 and 1, where the ratio is equal to 1.0 for a perfectly straight fiber.

Pulp preparation

Pulps were prepared from the various fiber length fractions with 0, 20 and 40 mg/g added CMC. The grafted amounts of CMC determined from conductometric titrations are given in Table 1. Adsorption was close to 20 mg/g for the dosage level of 20 mg/g and in the range of 27–40 mg/g for the 40 mg/g dosage level. The reason for the lower adsorption efficiency for the 40 mg/g dosage level is not known, but as will be shown, the less than ideal adsorption efficiency does not influence the stiffness and strength properties of the papers examined here. Reference to the different CMC treatments will be in terms of their CMC dosage levels, i.e., 0, 20 and 40 mg/g.

In order to set the pulp in its sodium counter-ion form, the pulp was immersed in 0.01 M HCl and the pH was adjusted to 2. The pulp was then soaked for 30 min and thereafter filtered and rinsed with deionized water. After this first washing stage, the pulp was transferred to its sodium counter-ion form by soaking it in 10^{-3} M $NaHCO_3$ at pH of 9, for 30 min. Thereafter, the pulp was filtered and repeatedly rinsed with deionized water until the conductivity of the filtrate was below 5 μS/cm.

The pulp was subsequently diluted to a consistency of 2.5% and was then heated to 120 °C for 2 h in a pressurized vessel at 1490 mm Hg. The added heat is catalyzing the attachment reaction of CMC onto the pulp fibers. Washing with deionized water followed this until the conductivity of the filtrate was below 5 μS/cm. Finally, the pulp was transferred to its hydrogen form, followed by a 2-h leaching of the pulp into its sodium form in order to remove excess CMC, not attached to the fibers.

Table 1 Grafted amount of CMC

Chip thickness, mm	CMC dosage, mg/g	Grafted amount of CMC, mg/g
1	20	20
	40	27
2	20	20
	40	33.8
3	20	18.6
	40	30.2
4	20	20
	40	38
5	20	20
	40	40
2–8 (Reference)	20	20
	40	40

Sheet forming

Isotropic sheets were formed from each set of pulp in a Finnish sheet former according to the SCAN-CM 64:00 [25]. Sheets of grammage 80 g/m^2 were manufactured for the in-plane tensile and fracture tests and 120 g/m^2 for the *Z*-direction tensile test. Wet pressing was performed in two ways: (1) low wet pressing—3.4 bar for 5 min, and (2) high wet pressing—a pressure of 3.4 bar was applied for 5 min, but after the first pressing the blotter papers were changed, and pressing was done once more the same way. After wet pressing, the sheets were inserted between drying plates and dried between dry blotters in a conditioning cabinet. The sheets were restrained from shrinkage by adhesion to the drying plates.

Tensile and fracture testing

Z-direction tensile strength was evaluated according to SCAN-P80:98, [26]. This test measures the through-thickness tensile strength of paper, defined as the failure load divided by the cross-sectional area. This test subjects fiber–fiber bonds to tension and provides an indication of the fiber–fiber adhesion. The test specimen is in the form of a circular sheet of paper which is adhesively bonded to metal platens, and the specimen is loaded in tension until it fails. It was necessary to improve the adhesive bond strength for testing of the strong CMC-treated papers. We replaced the double adhesive tape proposed in the standard by a strong heat-sensitive photo-mounting tissue; see Panek et al. [22]. The adhesive bonds between the metal blocks and the paper were achieved by placing the adhesive layer/paper/grip assembly in an oven under pressure. After bonding, the assembly was conditioned at 23 °C and 50% RH for 12 h. Ten replicate specimens were tested for each fraction.

In-plane tensile testing was conducted according to SCAN-P67:93, [27]. Specimens of 15 mm width were clamped between the grips of a tensile test machine and loaded to failure at a cross head rate of 1.7 mm/s. Ten replicate specimens were tested for each fraction. The tensile strength is defined as the maximum force, F_{ULT}, divided by the cross-sectional area A, of the specimen,

$$\sigma_T = \frac{F_{\mathrm{ULT}}}{A} \tag{1}$$

where $A = bt$, b being the width, and t the thickness of the paper specimens. The thickness of paper, however, is difficult to measure due to the soft non-uniform porous structure of the material; for this reason, the strength is divided with the density of the paper, SCAN-P88:01, [28]. The density is calculated from,

$$\varrho = \frac{W}{t} \tag{2}$$

where W is the areal weight (grammage) of the paper (kg/m^2) and t is the thickness, also defined in SCAN-P88:01, [28]. By combining Eqs. (1) and (2), we obtain the specific strength,

$$\frac{\sigma_T}{\varrho} = \frac{F_{\mathrm{ULT}}}{bW} \tag{3}$$

It is observed that the specific strength calculation does not require determination of the sheet thickness. The specific strength is called "tensile index," with

units Nm/kg. In addition to measuring the tensile strength, specific elastic modulus (E/ϱ) and strain to failure (ε_T), it is possible to determine the tensile energy absorption, which is the area, U, under the load–elongation curve (Nm). Similar to the tensile strength, a tensile energy absorption index with units Nm/kg or J/kg is obtained by first dividing U with the volume of the test specimen gage region ($V = blt$), where l is the gage length, and then dividing the results by the sheet density,

$$\frac{U}{V\varrho} = \frac{U}{blW} \tag{4}$$

Here, U is the area under the load–displacement curve, V is the volume of the specimen under load, ϱ is the density of the sheet, b is the specimen width, l is the gage length, and W is the grammage.

In-plane fracture mechanics testing was conducted according to SCAN P77:95, [29]. A rectangular test specimen with a central sharp notch of length $2a$, as shown in Fig. 2, is loaded in tension until the specimen fails by crack propagation. The standard specifies a 50-mm-wide specimen with a 20-mm central notch. Twelve replicate fracture specimens of each fiber length fraction were prepared and tested. It has been observed that the cracked region of the fracture test specimen tends to buckle transverse to the loading direction due to a non-uniform Poisson contraction. To minimize the influence of this undesired deformation mode, an anti-buckling guide was used to cover the center of the specimen; see the test standard. The analytical foundation for the J-integral standard, SCAN P77:95 [29], is outlined in a paper published by Wellmar et al. [38]. They present nonlinear elastic–plastic finite element analysis of the center notch specimen, as shown in Fig. 2, to calibrate an analytic nonlinear solution for the plastic part of the J-integral. Critical value of J-integral, i.e., the fracture toughness, J_c, is calculated from the measured fracture load using a formula involving both the elastic and plastic contributions to J_c provided in this standard. The basic units of J_c are J/m^2. Dividing J_c with the density yields a property, called "fracture toughness index" with units of Jm/kg. All mechanical tests were conducted in a controlled laboratory environment at 23 °C and 50% RH.

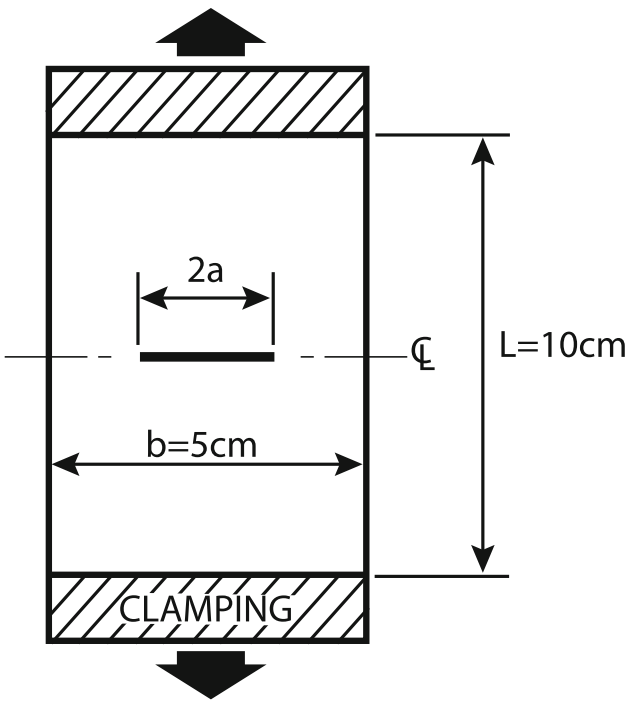

Figure 2 Center-notch specimen for fracture testing.

Results and discussion

Fiber length distributions for the various fractions are shown in Fig. 3. The thicker chips and reference set of chips provide higher averages and wider distributions. Preparation of pulp inevitably produces very short fiber fragments "fines," as shown in Fig. 3. The thicknesses of the 1- and 2-mm-thick chips are less than the natural fiber length which results in narrow distributions of fiber length, as shown in Fig. 3. As mentioned earlier, the length of spruce fibers is about 2.89 mm in average [7]. Chips thicker than about 3 mm are not expected to provide a significant amount of longer fibers and wider distributions of fiber lengths, as shown in Fig. 3.

Table 2 summarizes the average fiber length, fiber shape factor and fines content determined by TAPPI T261 cm-10 [33] for the various fractions. The shape factor is close to unity for all fractions indicating straight fibers. It is noted that the fines content is quite small for all fractions. Based on the data in Tables 1 and 2, the fibers in the different fiber length fractions are considered similar with regard to chemical composition. Hence, the produced fiber material was judged adequate for this study.

Figure 4 shows the sheet density for all fiber length fractions plotted versus the amount of CMC addition. The results show no significant influence on the density by CMC addition. The sheet density was

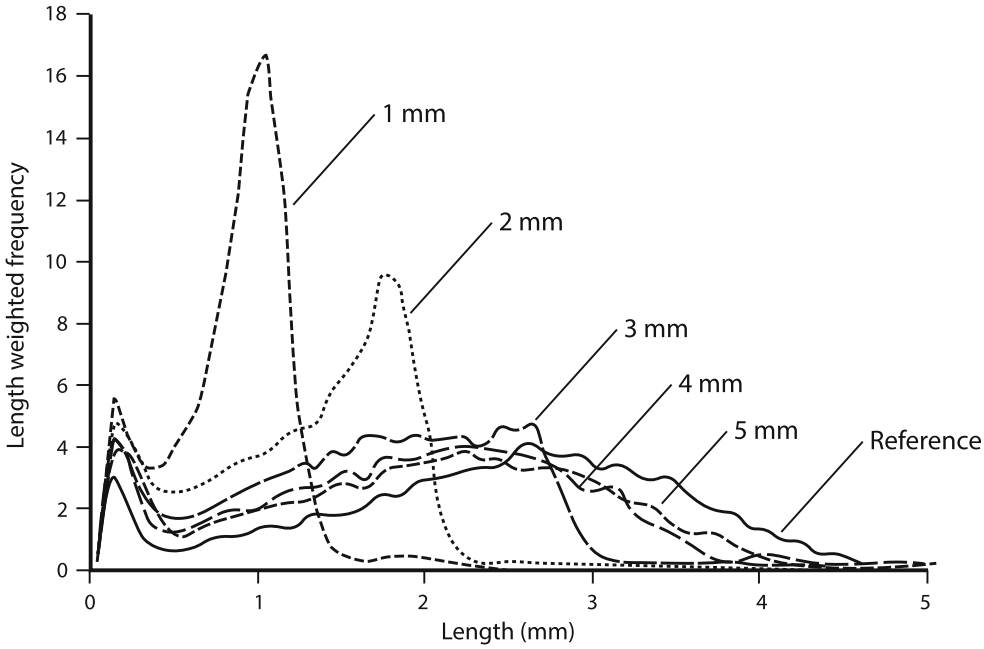

Figure 3 Fiber length distribution for the various chip thicknesses.

Table 2 Data for the different fiber length fractions

Chip thickness, mm	Fiber length, mm	Shape factor	Fines content, %
1	0.87	0.88	5.0
2	1.33	0.89	3.5
3	1.73	0.90	3.1
4	2.02	0.91	2.8
5	2.06	0.92	1.6
2–8 (Reference)	2.55	0.86	2.5

The length is the weighted arithmetical mean

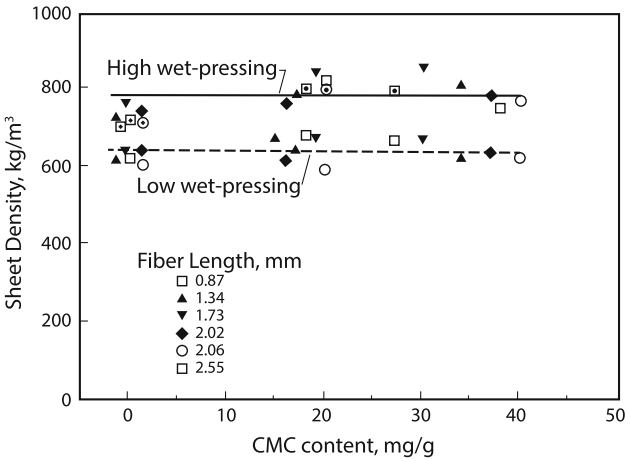

Figure 4 Sheet density versus CMC content for different fiber lengths. Results are shown for two wet pressing levels.

about 650 kg/m^3 for low wet pressing and close to 800 kg/m^3 for high wet pressing. The main objective of this investigation is to examine the effects of fiber length and fiber–fiber bond strength on tensile strength and toughness properties of paper. Such properties depend on porosity which is indicated by the sheet density. A common method to compensate for this factor is to divide the stiffness, strength and toughness by the sheet density to obtain "specific" properties. Still, also the specific properties depend on sheet density. To account for the difference in density due to wet pressing conditions, as shown in Fig. 4, it was decided to reference the results (specific modulus, specific strength and specific toughness) to a reference, intermediate density of 700 kg/m^3. The specific stiffness, strength and toughness for this reference density were obtained by linear interpolation of the results for low and high wet pressing. Such a procedure is considered reasonable due to the small difference in density for the two wet pressing conditions.

Z-directional tensile strength results for the untreated and CMC-treated papers pulps with fiber lengths of 0.87 and 2.55 mm are listed in Table 3. The scatter in strength ranged from 2 to 4%. The results for the other fiber length fractions are similar. CMC addition of 20 mg/g greatly improved the Z-strength compared to the untreated papers. CMC addition of 40 mg/g did not provide much additional improvement of strength.

The tensile stiffness is determined from the initial slope of the stress–strain curve in the elastic region. The controlling mechanisms for the elastic modulus of paper have been examined by Page et al. [21]. The starting point for such analysis is the theory of Cox [3]. He initially assumed that the strain in a sheet of paper is uniform so that the stress in each fiber may be calculated from the stress transformation equation, Timoshenko and Goodier [32]. Such analysis yields the specific sheet modulus, in terms of specific fiber modulus [3],

$$\frac{E}{\varrho} = \frac{E_f}{3\varrho_f} \tag{5}$$

where E_f and ϱ_f are the fiber modulus and fiber density. Page et al. [20] measured fiber modulus for softwood fibers and found values of about 45–55 GPa. With a fiber wall density of 1500 kg/m^3, [24], Eq. (5) provides a sheet modulus in the range from 10 to 12 MNm/kg. Such high values may be realized for papers with well-bonded straight fibers pressed to high densities, but crimps and bends in fibers, and lack of bonding (RBA) tend to reduce the modulus. Cox [3] explained this deviation in terms of non-uniform strain in short fibers which justified the shear-lag concept.

The tensile stiffness index is plotted versus fiber length in Fig. 5. Scatter in the test data ranged from 3 to 5%. For the CMC-treated papers, the tensile stiffness does not depend on fiber length. The tensile stiffness index is about 7 MNm/kg, not far from the theoretical upper bound value discussed above, (10–12 MNm/kg). For the untreated papers, the specific modulus is much smaller, but shows an increasing trend with fiber length. This increase appears to be due to the shear-lag effect discussed by Page et al. [21]. As discussed in Introduction, the addition of CMC also disperses the fibers and improves the sheet formation [35, 36]. Such effects are most likely causing the stiffness increase in the CMC-treated papers.

Table 3 *Z*-directional tensile strength (MPa) for papers with 0.87-mm fibers and reference fraction

CMC, mg/g	0.87 mm	Reference (2.55 mm)
0	0.30 ± 0.001	0.27 ± 0.001
20	1.00 ± 0.04	0.77 ± 0.03
40	1.10 ± 0.04	0.90 ± 0.04

Reference density = 700 kg/m^3

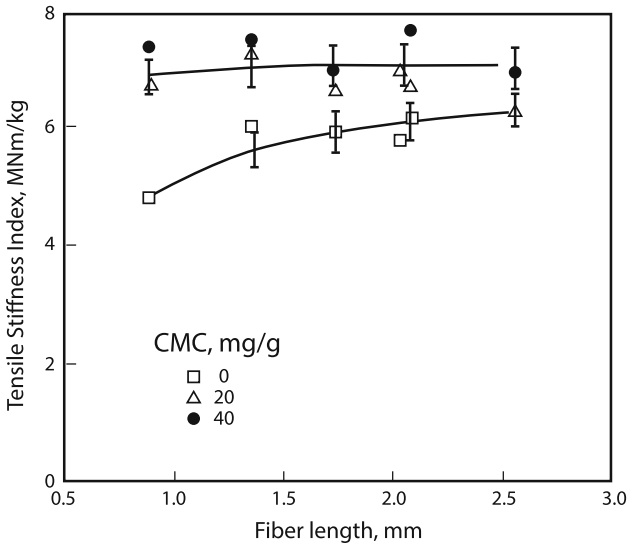

Figure 5 Tensile stiffness index versus fiber length for different levels of CMC addition.

The tensile strength (tensile index) is plotted versus fiber length in Fig. 6. The scatter ranged from 4 to 5%. For the untreated specimens, the tensile strength increased (by 82%) over the range of fiber lengths

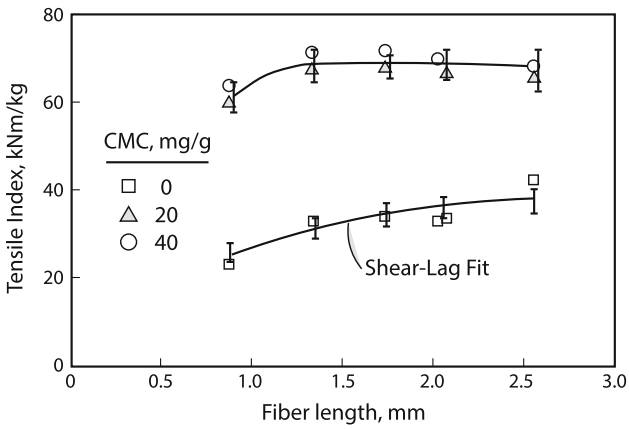

Figure 6 Tensile strength index versus fiber length for different levels of CMC addition. The curve for the untreated papers is a fit to the shear-lag model.

examined. The addition of CMC increased the strength substantially for all fiber lengths, to a level independent of fiber length. The results for the untreated paper may be compared to shear-lag model predictions [2]. For a paper with fibers longer than the critical length, l_c, this model predicts the tensile index

$$T_s = T_0\left(1 - \frac{l_c}{2l}\right) \tag{6}$$

where T_0 is the tensile index for a paper with fibers so long that pull-out failure can be neglected, and l is the actual fiber length.

The shear-lag model, Eq. (6), was fitted to the data for the untreated papers in Fig. 6. A least squares fit provided: $T_0 = 47$ kNm/kg, and $l_c = 0.87$ mm. The fitted equation is indicated as the curve in Fig. 6. Extrapolation of the results to infinite fiber length, $l \to \infty$, provides a strength of 47 kNm/kg, which is quite far below upper limit strength of the CMC-treated papers ($T_0 \sim 70$ kNm/kg). Hence, even if infinitely long untreated fibers were hypothetically used, the strength of the untreated paper would fall below the asymptotic strength of the CMC-treated papers. As discussed earlier, CMC addition not only improves bond strength, but also helps to disperse the fibers and improve sheet formation, Yan et al. [35, 36]. This could possibly explain the very large strength elevation due to CMC addition observed in Fig. 6.

The results for the tensile strain at break are shown in Fig. 7. The scatter range was from 3 to 5% of the mean strain to failure. Increasing fiber length results in larger strain to failure. Addition of CMC provides substantially higher failure strains for all fiber lengths. The strain to failure provides an indication of ductility of the material, and the results in Fig. 7 indicate that addition of CMC should provide tougher papers.

The tensile energy absorption is plotted versus fiber length in Fig. 8. The scatter was 6–8%. The trends of tensile energy absorption versus fiber length are similar to those for tensile strength (Fig. 6) and strain to failure (Fig. 7). For the untreated papers, the tensile energy absorption increases with fiber length, while the CMC-treated papers display a high level at all fiber lengths. The tensile energy absorption results for the untreated papers in Fig. 8 agree qualitatively with predictions from the shear-lag analysis of Kelly [13] pointing to frictional sliding

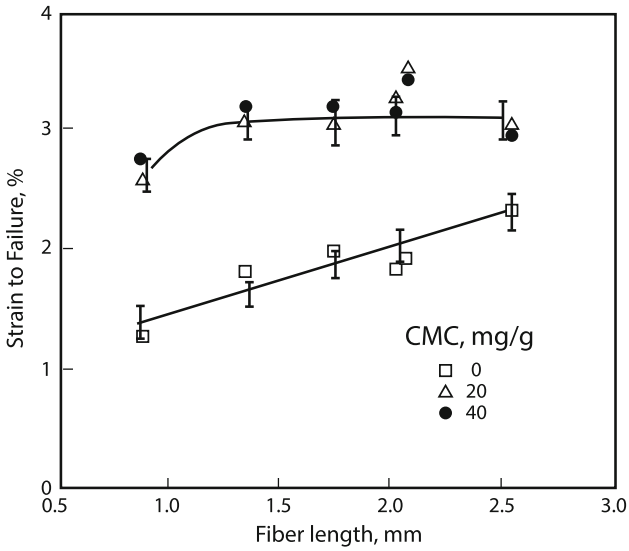

Figure 7 Strain at break versus fiber length for different levels of CMC addition.

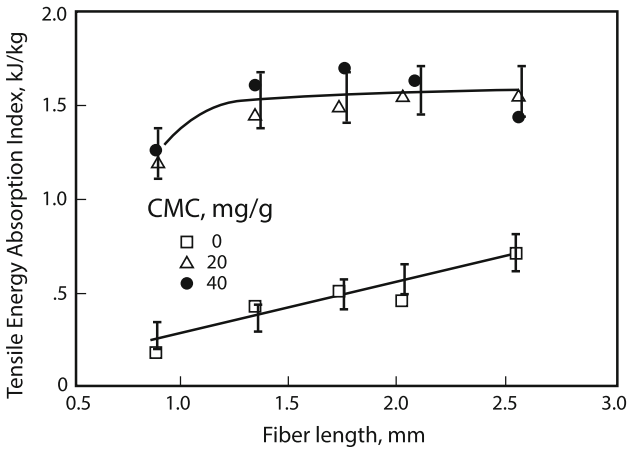

Figure 8 Tensile energy absorption index versus fiber length for different levels of CMC addition.

work upon pull-out of debonded fibers as an energy-absorbing mechanism. For the CMC-treated papers, however, the results seem to indicate that fracture of fibers dominates the tensile energy absorption.

The fracture toughness for the untreated and CMC-treated papers is plotted versus fiber length in Fig. 9. The scatter in this property ranged from 6 to 8%. The fracture toughness increases strongly with fiber length for both the untreated and CMC-treated papers. This is in contrast to the results for the tensile energy absorption shown in Fig. 8, which shows a small dependence on fiber length for the CMC-treated paper. Notice though that fracture toughness characterizes the resistance to propagate a sharp

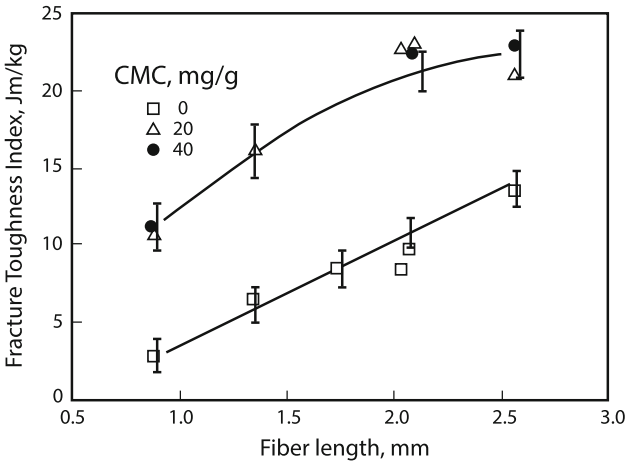

Figure 9 Fracture toughness index versus fiber length for different levels of CMC addition.

crack, while the tensile energy absorption is determined from testing of a specimen without a crack. The results in Fig. 9 show that CMC addition is an extremely beneficial method to improve fracture toughness, especially for papers with short fibers, where the toughness is increased roughly by a factor four. The results also show that the results for 20 and 40 mg/g CMC addition are very similar, indicating 20 mg/g is sufficient. It is possible that smaller amounts of CMC addition would be sufficient.

Conclusions

Fiber length and fiber-to-fiber bonding effects on tensile properties and fracture toughness of kraft paper have experimentally been investigated. The tensile strength and fracture work of papers with weak fiber–fiber bonds increased with fiber length consistent with the shear-lag model. CMC-treated papers with strong bonds displayed high strength and work of fracture independent of fiber length which indicates that the failure process is governed by fiber failures rather than bond failures, even at short fiber lengths. The fracture toughness, expressed as the critical value of the J-integral, increased strongly with fiber length for both untreated and CMC-treated papers. The results show that long fibers and CMC addition are extremely beneficial for improving the fracture toughness.

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