Comparison of fracture toughness of paper with tensile properties

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SUMMARY

The paper web runnability is significantly affected by the presence of defects in the material. Fracture toughness (FT) of paper measures its resistance to fracture from pre-existing flaws and is closely related to the runnability. However, lack of a rapid and reliable technique to measure FT is a major drawback in the improvement of runnability. The aim of this work was to find any relationship between conveniently obtainable tensile properties (tensile strength, elastic modulus and Tensile Energy Absorption (TEA)) and FT. Twelve samples were measured, from a mixture of machine-made papers and handsheets. The range of FT measured by the Essential Work of Fracture (EWF) method was 8.2 to 28.9 J.m/kg. In this work, tensile index and TEA were both found to be linearly correlated with FT with correlation coefficients of $R^2 = 0.72$. A combination of tensile index and TEA gives a better correlation with fracture toughness with $R^2 = 0.84$. However in all cases the correlation was not strong enough to justify using either tensile properties or a combination of properties to predict the fracture toughness.

Keywords
Fracture toughness, essential work of fracture, DENT geometry, tensile energy absorption, Moving Belt Drainage Former (MBDF)

Fracture toughness (FT) is undoubtedly one of the important fundamental mechanical properties of any material. FT measures the resistance offered by a material against the propagation, under stress, of cracks from existing defects within the material. FT is also a key mechanical property for many grades of paper, since it is closely related with paper web runnability. 'Runnability' is the term commonly used in the paper industry to describe the efficiency of paper web production or end use conversions. Paper web breaks severely impair the efficiency of both on-machine operations and end use operations. The requirement to improve the web efficiency by minimising breaks remains a key task in paper technology research.

Defects in paper can include shives, edge tears, stickies, calender scabs, hairs, holes and plastic or metal contaminants. These defects can behave as cracks. Although there have been significant advances made in the past five decades in the reduction of paper web failures by adopting new screening techniques, on-line defect monitoring and careful process control, paper web failures are still common in the industry. One of the major drawbacks in the improvement of FT is the lack of a well-established technique that can be applied to rapidly assess the FT of paper.

Paper is a visco-elastic material and can show significant plastic deformation under plane stress. The direct application of Linear Elastic Fracture Mechanics (LEFM) to calculate the FT is limited to cases where only small-scale yielding is observed. The application of LEFM to paper (*1*) was unsuccessful since the plastic zone is not limited to a small area in front of the crack tip (called the Fracture Process Zone, FPZ) but expands to a much larger region.

Non-linear fracture mechanics methods, such as the J-integral, and energy methods, such as Essential Work of Fracture (EWF), that were developed to estimate the FT of ductile materials, have also been applied to paper (*2,3*). The J-integral method based on multiple specimens requires measuring load-elongation curves for a series of notched specimens with different crack lengths followed by graphical interpretation to obtain the FT. For these reasons the multiple-specimen J-integral technique is not feasible for routine measurements. The single specimen technique, which successfully overcomes the above problems, is associated with other problems. Some of the assumptions made in this technique are not always true and in some cases the estimated FT depends on sample or crack geometry (*4,5*) and is not an intrinsic property of the material. Based on a J-integral type method, an instrument has been developed to measure paper FT (*6,7*). This requires the measurements of both notched and unnotched samples at a single sample size to calculate the FT. A number of parameters need to be obtained by fitting the data from the unnotched samples before the FT can be calculated. Karenlampi (*8*) and his co-workers compared the two test methods, J-integral and EWF, for estimating the FT and observed some clear differences in the measurements for tough ductile papers, where the J-integral method seemed to underestimate the FT. For these reasons the reliability of the J-integral method in the application of estimating the FT of paper is questionable.

In comparison to the single specimen J-integral technique, the EWF method requires a large number of samples of differing sizes be measured, consuming a significant amount of time to determine a single FT value. In some instances the assumptions underlying the EWF technique are not always met either (*9,10*). However, the EWF method has earned some respect in terms of its reliability and measures the FT as an intrinsic property of the material.

Techniques for the measurement of the tensile properties of paper, such as tensile strength, elastic modulus and Tensile Energy Absorption (TEA), are well established and straightforward. If a correlation between fracture toughness and the tensile properties could be established, then it might be possible to use one of these properties to predict the fracture toughness.

Although previous studies showed some correlation between tensile properties and FT (*3*), no attempts were made to quantify the correlation, especially between TEA – FT. This study is an attempt to find a correlation between FT and the tensile properties of paper. The EWF technique has been used as a measure of FT. Various commercial papers and

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laboratory-made handsheets have been used in this investigation.

EXPERIMENTAL METHOD AND MATERIAL

Essential work of fracture theory

Following initial work by Broberg (*11*), Cotterell and Reddell (*12*) developed the EWF technique. The basic idea behind this technique is that the failure zone in front of a crack is contained within a process zone. This zone is subdivided into two smaller zones. The zone where the crack propagates (Fracture Process Zone, FPZ) and a surrounding zone where plastic deformation occurs (outer plastic zone). Although the energy consumed in the FPZ is essential to the fracture, the energy consumed in the outer plastic zone is not. The total work (W_f) estimated from an area under a load-elongation curve of a notched elastic-plastic material contains both essential and non-essential work. The EWF method separates the total work into the essential work of fracture (W_e) and the plastic work (W_p) of a sample prepared to Double-Edge Notched Tension (DENT) geometry. The DENT geometry consists of notches, where fracture is initiated, cut either side of a central ligament of length L. In this method, it is assumed that $W_e \propto L$ and $W_p \propto L^2$ (*12*). Under these assumptions, we can write,

$$w_f = w_e + \beta L w_p \qquad [1]$$

where $w_f = W_f/Lt$, t is the sample thickness, w_e is the specific essential work of fracture (the FT) and β is a function of specimen geometry.

The FT is estimated by measuring W_f at a number of different ligament lengths, plotting w_f versus L and fitting a straight line to the data to determine the y-axis intercept, which is the FT, w_e. However, for linearity to hold, suitable ligament lengths have to be selected such that the stress field across the ligament remains uniform before failure (*13*). When applying the EWF technique to paper, sample thickness (t) is replaced by the sample grammage (mass per unit area,g/m^2) to minimise the error contributed by the non-uniformity of a sample.

Materials

The following commercial papers were used for the EWF and tensile tests: (a) 'Reflex' copy paper (MD and CD), (b) plaster linerboard (MD and CD) and (c) sack kraft (MD and CD). The following laboratory-made handsheets were also tested: (d) bleached radiata pine kraft pulp (New Zealand) of medium coarseness, prepared at different beating and pressing levels.

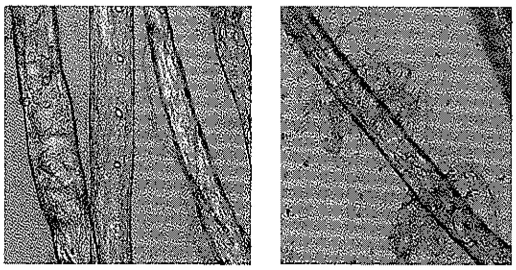
Handsheet preparation and processing

The dried, bleached New Zealand radiata pine, market kraft pulp, which was categorised as medium coarseness (*14*), was used for the handsheet preparation. The average fibre length of this pulp was given as 2.24 mm.

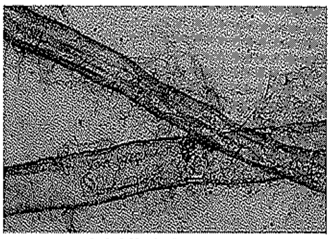
Pulp samples were soaked in distilled water for about five hours before being disintegrated using a valley beater and a standard disintegrator. The valley beater was also used to refine the fibres. The beating was carried out according to ISO-5264/1-1979. One set of handsheets was made without any beating and another two sets were prepared, with medium and high beating levels, using the Moving Belt Former (MBDF). Xu and Parker (*15*) have given a detailed description of the MBDF construction and operation. The initial freeness of the pulp (before beating) was about 720 CSF. After 30 minutes beating the freeness was reduced to 630 CSF. The pulp taken for handsheet preparation at this stage was named 'medium beaten' pulp. The pulp beaten for 60 minutes was categorised as 'highly beaten' pulp and had a freeness of 460 CSF.

Figure 1 (a), (b) and (c) show optical microscopic images of unbeaten, 30 minutes beaten and 60 minutes beaten radiata pine respectively. The optical micrographs in Figure 1 (b) and (c) clearly show the torn fibre surface and partially removed fibrils resulting from the mechanical action in beating.

Wet pressing is another important parameter that can influence the mechanical properties of paper. The 'Fibre Tech' sheet roll press was used for the wet pressing. In this work two pressing levels, one with low pressure (200 kPa) and another with relatively high pressure (600 kPa) were used.



(a) Unbeaten (b) Medium beaten



(c) High beaten

Fig. 1 **Micrographs of (a) unbeaten, (b) medium beaten, and (c) highly beaten radiata pine kraft pulp fibres.**

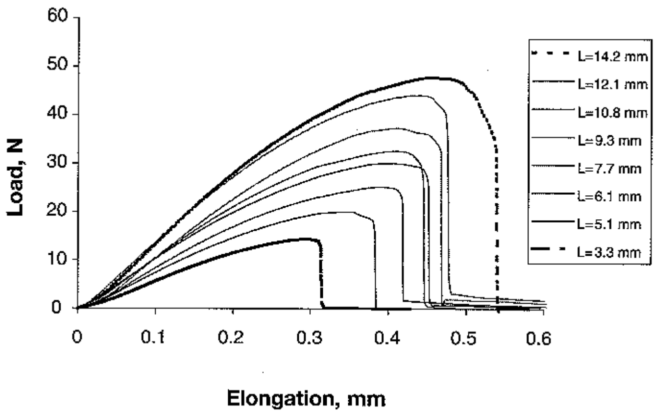


Fig. 2 **Load – elongation curves obtained from 'Reflex' copy paper (MD) DENT samples at different ligament lengths.**

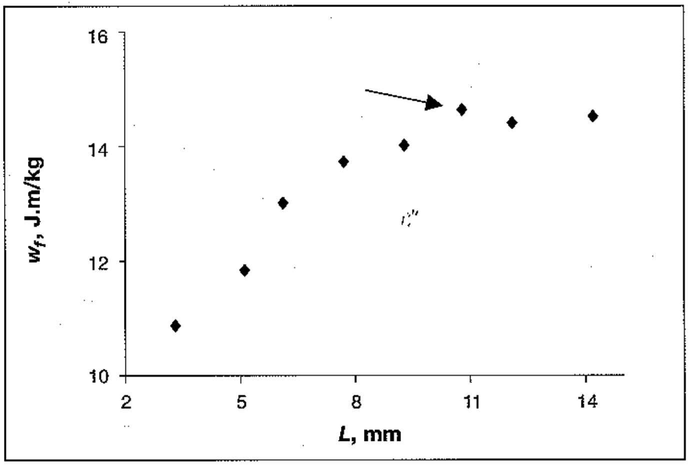


Fig. 3 Plot of w_f against ligament length (L) for 'Reflex' copy paper (MD).

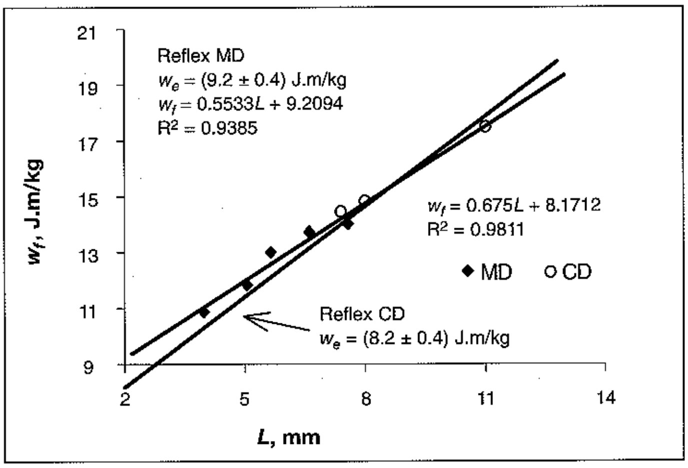


Fig. 4 Plot of w_f against L for 'Reflex' copy paper. The two straight lines are linear fits to individual MD and CD data sets.

Physical testing

Uniform DENT sample preparation is one of the greatest difficulties in the use of the EWF technique. For this work a special cutting die was designed to provide multiple ligament lengths from a 220 by 220 mm handsheet. A self-release, GSB-1 series hydraulic swing beam cutting press was used to stamp out uniform test pieces using this die.

All the samples were conditioned in accordance with ISO 187 and all the test pieces were prepared and tested in the same standard atmospheric conditions.

FT tests were carried out on DENT samples, using a specially designed pair of line type clamps (*3*) mounted on an Instron model 5566 universal testing machine. The prepared DENT samples were 90 mm long. The dimensions of the samples were selected to satisfy one of the EWF conditions, $(B/3) > L$, where B is the width of the sample (*12*). The crosshead speed of the Instron was set at 2 mm/min.

Tensile properties

The tensile properties such as tensile index, elastic modulus and tensile energy absorption were measured according to ISO 1924-2:1994 using the Instron universal tester.

RESULTS AND DISCUSSION

EWF results – commercial papers

Reflex copy paper (MD and CD): The diagram in Figure 2 shows representative load-elongation curves obtained for different ligament lengths of 80 g/m² Reflex copy paper in the Machine Direction (MD). The curve with the largest area under the load-elongation plot represents L=14.2 mm, while the smallest represents L=3.3 mm. The apparent thickness and density of these samples were 102 μm and 784 kg/m³ respectively. At least 15 DENT samples were tested for each ligament length L. The average area under each load-elongation curve was taken for the final w_f calculation. The graph in Figure 3 shows the values of w_f against L for eight different ligaments lengths.

To determine the FT (w_e) of the copy paper in the MD direction, the y-axis intercept of the data in Figure 3 must be determined through extrapolation. However, the last 3 data points in the above figure (see arrow) with $L > 10$ mm do not follow this line. The most likely reason for the above behaviour could be that when $L > 10$ mm the sample has not completely yielded before the crack propagates (*9*). Hence DENT test pieces with $L > 10$ mm are not suitable to use with MD copy paper samples when applying the EWF method. A similar behaviour was observed for CD copy paper samples, where the data obtained for $L \geq 17$ mm did not fall on the same straight line as the rest of the data. Figure 4 shows the w_f against L data fitted to a linear relation for tests conducted on copy paper in the MD and

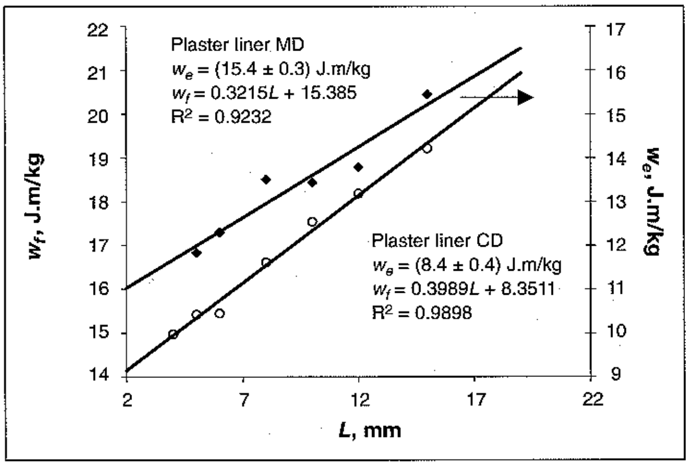


Fig. 5 Plot of w_f against L data sets and the respective linear fittings to plaster liner for MD and CD tests.

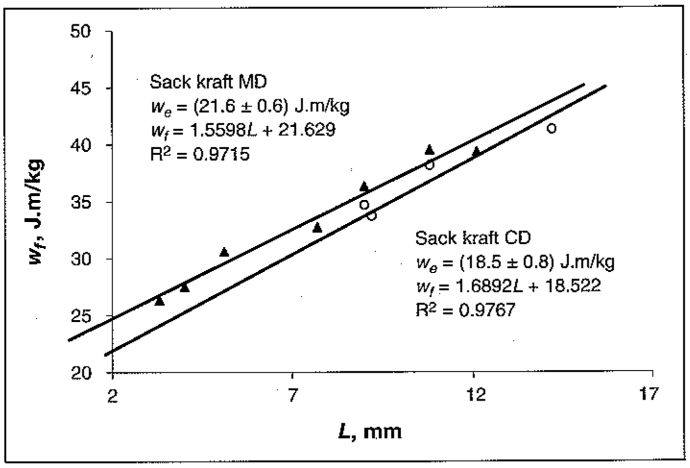


Fig. 6 Plot of w_f against L data for sack kraft (MD and CD).

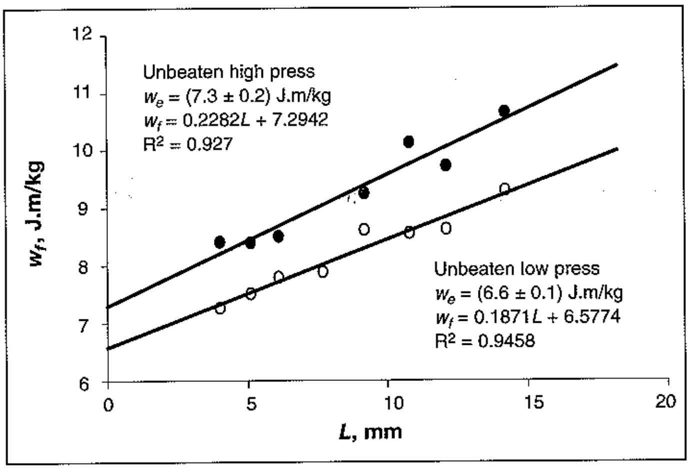


Fig. 7 Plot of w_f against L data for low and high wet-pressed unbeaten radiata pine kraft pulp.

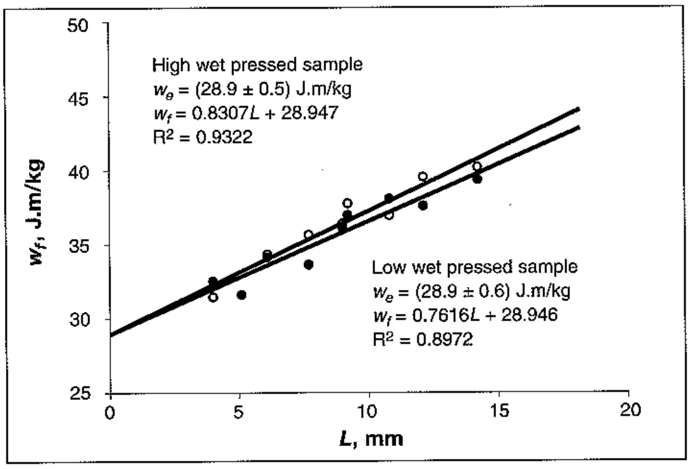


Fig. 8 Plot of w_f against L data for low and high wet-pressed medium beaten radiata pine kraft pulp.

CD direction. The data obtained for $L > 10$ mm for testing in the MD direction and $L \geq 17$ mm for the CD direction were omitted from the fitting.

The fracture toughness determined from the y-intercepts in Figure 4 is 9.2 ± 0.4 J.m/kg in the MD direction and 8.2 ± 0.4 J.m/kg in the CD direction. The larger slope for CD compared to that for MD reflects its greater extensibility.

Plaster linerboard (MD and CD): Figure 5 shows the w_f against L data obtained for 190 g/m² plaster linerboard in MD and CD directions. The mean thickness and density of these samples were 310 μm and 613 kg/m³ respectively. The estimated FT for plaster liner is 15.4 ± 0.3 J.m/kg for the MD direction and 8.4 ± 0.4 J.m/kg for the CD direction. The FT for the CD direction is comparable to that of copy paper in the CD direction. However, unlike the case in copy paper where the FT of MD and CD differed only by 11%, FT along CD for plaster liner was only about half of the value of that in the MD.

Sack kraft (MD and CD): The grammage of the tested samples was 69 g/m². The thickness and density were 163 μm and 491 kg/m³, respectively. Figure 6 shows w_f against L for sack kraft for the MD and CD directions. The estimated FT for the MD and CD directions are 21.6 ± 0.6 J.m/kg and 18.5 ± 0.8 J.m/kg, respectively.

EWF results – laboratory made handsheets

Unbeaten radiata pine: The grammage of the prepared handsheets was 76 g/m². The mean thickness and density of low wet-pressed samples were 183 μm and 415 kg/m³, respectively.

The thickness and density of the high pressed samples were 156 μm and 487 kg/m³ respectively. Figure 7 shows the data obtained for unbeaten radiata pine for the two different pressing levels. The FT estimated for the low pressed handsheets was only 6.6 ± 0.1 J.m/kg. The handsheets pressed with 600 kPa pressure show a slightly higher FT of 7.3 ± 0.2 J.m/kg. This indicates that the better sheet consolidation and fibre compaction, which resulted from the wet pressing, has improved the fracture toughness.

Medium beaten radiata pine: The fracture toughness of the medium beaten radiata pine was 28.9 J.m/kg, independent of the degree of pressing (see Fig. 8). The small increase in the slope of the high wet pressed sample compared to that of the lightly pressed was discounted as an effect of wet pressing, since the difference is within the experimental uncertainty.

Highly beaten radiata pine: Figure 9 shows the w_f against L plots for the highly beaten medium radiata pine. The FT obtained for high and low wet pressing were very similar with 26.6 J.m/kg for low pressed samples and 26.9 J.m/kg for the high pressed samples. The difference in w_e of these two measurements is within the uncertainties. The FT of the highly beaten samples is ~8% less than for the

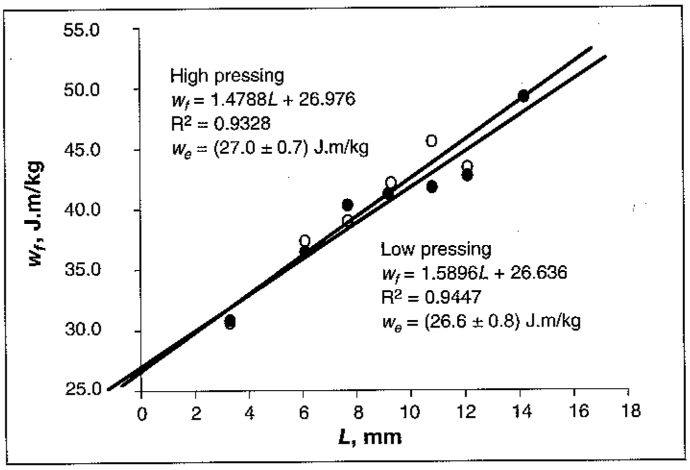


Fig. 9 Plot of w_f against L data for low and high wet-pressed highly beaten radiata pine kraft pulp. The straight lines are linear fits for the respective data sets.

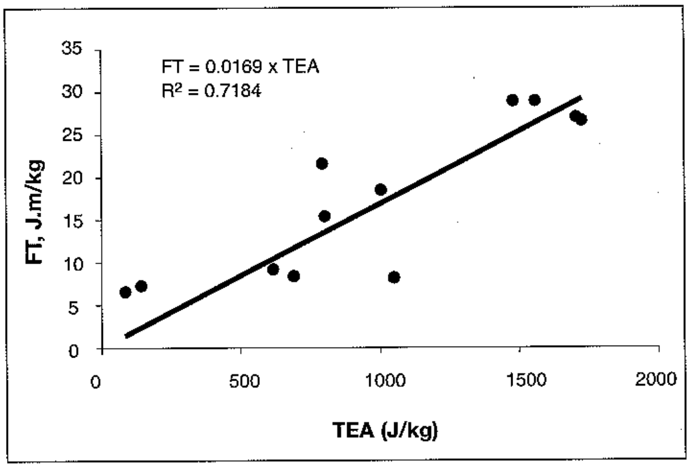


Fig. 10 Fracture toughness (FT) against TEA index for test samples.

Table 1

Summary of tensile properties (L.Pres = low pressing, H.Pres = high pressing).

		Tensile index (N.m/g)	Elastic modulus (kN.m/g)	TEA index J/kg
Reflex	MD	52.0 ± 1.3	6.9 ± 0.3	617 ± 27
copy	CD	34.4 ± 1.3	4.4 ± 0.2	1049 ± 46
Plaster	MD	58.1 ± 1.1	6.2 ± 0.1	802 ± 44
liner	CD	22.3 ± 0.5	2.6 ± 0.1	691 ± 45
Sack	MD	63.9 ± 3.7	7.0 ± 0.3	793 ± 65
kraft	CD	34.4 ± 1.3	3.8 ± 0.1	1004 ± 139
Pine	L.Pres	14.9 ± 0.5	2.9 ± 0.1	85 ± 4
unbeaten	H.Pres	19.0 ± 0.3	3.4 ± 0.1	143 ± 9
Pine	L.Pres	62.1 ± 4.8	7.2 ± 0.4	1480 ± 29
med. beat	H.Pres	64.63 ± 4.9	6.7 ± 0.3	1560 ± 33
Pine	L.Pres	69.1 ± 2.6	6.8 ± 0.3	1707 ± 38
high beat	H.Pres	75.3 ± 1.3	7.7 ± 0.2	1726 ± 32

Table 2

Summary of the correlation between fracture toughness and tensile properties.

	R^2	Average error (%)	Maximum error (%)
FT-tensile index (T)	0.72	26	104
FT-TEA	0.72	34	116
FT-elastic modulus (E)	0.56	37	140
FT-T^2/E	0.74	30	70
FT-Tx $(TEA)^{1/2}$ x10^{-6}	0.84	30	78

medium beaten samples. The reduction in FT could either be due to fibre shortening or fibre weakening due to excessive beating.

Tensile properties

Table 1 shows the tensile properties of all the test materials. For the commercial papers, the tensile index and elastic modulus in the MD direction were all at least 50% greater than in the CD direction.

The TEA index has shown some mixed results for commercial papers, as 'Reflex' and sack kraft in the CD direction have shown larger TEA values than in the MD direction, in direct contrast to the FT results.

Figures 10, 11 and 12 show the graphs of FT against tensile index, TEA and elastic modulus respectively, for all twelve samples. The data in each graph was fitted with a linear function forced through the origin. Table 2 summarises the R^2 values obtained from the fits. Table 2 also lists average and maximum errors. These were determined for each tensile property by using the relevant fitted linear function to estimate the FT from each data point. The FT estimated from the tensile property was then compared with the measured FT to determine the average and maximum error.

A fairly reasonable relationship can be observed between FT-TEA and FT-tensile index with $R^2 \approx 0.72$. In addition a weaker relationship can be observed between FT - elastic modulus ($R^2 \approx 0.56$). If tensile index had been used to predict the FT then the average error would be 26%, while the maximum error was 104% for 'Reflex' copy paper in the CD direction. As can be seen from Table 2, the use of either TEA or elastic modulus to predict FT gave higher maximum and average errors than when using tensile index.

It might have been expected that the best correlation would have been between TEA and FT, because TEA is also a measure of energy. However in the CD and MD directions of 'Reflex' copy paper and sack kraft the correlation with FT is extremely poor, as the TEA in the CD direction is higher than in the MD direction, but the FT of these samples behaves oppositely. One reason for such behaviour lies in the anisotropy of the machine made paper, where the stress field at the fracture zone is dependent on the MD/CD anisotropy. Another reason is that the TEA includes contributions from the energy absorbed in fracture as well as plastic work in the rest of the sample. We cannot measure this plastic work directly, but it should be related to the slope, βw_p, determined from the EWF graph. The values of βw_p for the data presented in this paper have been plotted against the measured FT in Figure 13.

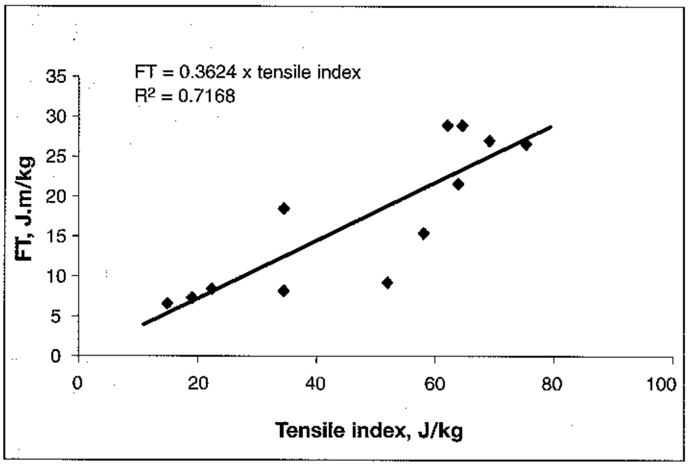


Fig. 11 Plot of FT against tensile index.

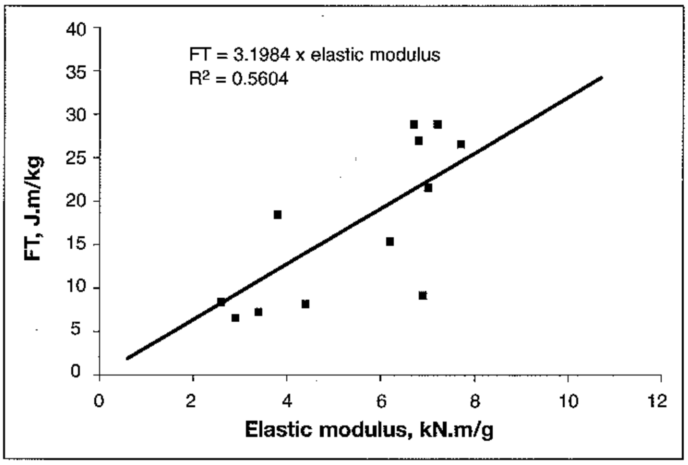


Fig. 12 Plot of fracture toughness (FT) against elastic modulus.

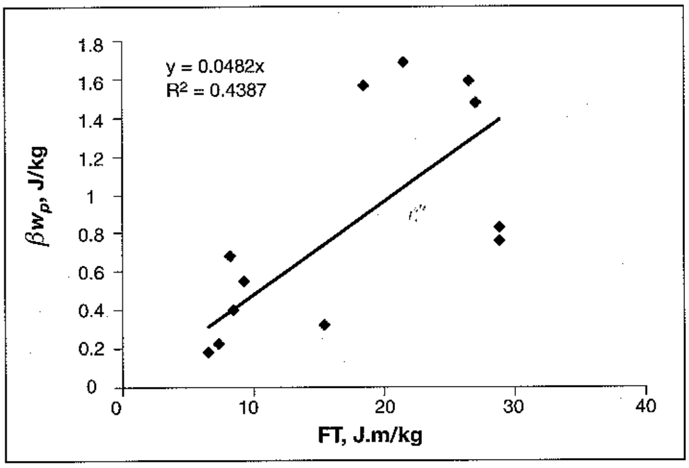


Fig. 13 Plot of βw_p against FT for all test samples.

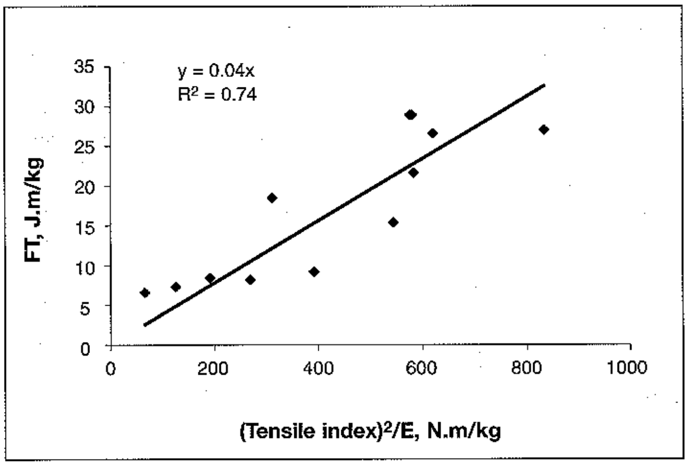


Fig. 14 Plot of fracture toughness against (tensile index)^2 /elastic modulus.

It can be seen that the correlation between plastic work (as estimated by βw_p) and FT is very poor, suggesting that the correlation between TEA and FT should also be poor.

Attempts were also made to find a correlation between fracture toughness and combinations of tensile parameters. In this work an attempt was made to establish a correlation between fracture energy, w_e, and a combination of tensile index and elastic modulus. A relation between these parameters was obtained by estimating the work from an area under a linear stress-strain curve of an elastic material. The approximate fracture energy of an elastic material under a stress-strain curve is $w_e = 1/2\sigma\varepsilon$, where σ and ε are stress and strain of the material respectively. Using Hooke's Law, where the elastic modulus E is related to stress and strain by $E = \sigma/\varepsilon$, the relation $w_e = {}^1\!/\!{}_2\sigma^2/E$ was obtained. For the work here the stress was replaced by tensile index and the fracture toughness against (tensile index)^2/(elastic modulus) graph was plotted as shown in Figure 14. The correlation coefficient obtained was 0.74. It was apparent that the tensile values in the MD direction of 'Reflex' copy paper and plaster liner were the least correlated with fracture toughness and removal of these values improved the correlation coefficient to $R^2 = 0.83$.

The work carried out by Uesaka et al., (*16*) on factors controlling paper runnability established an excellent relationship between web break frequency and (tensile strength) x (stretch)$^{1/2}$ for the MD direction. Since the web breaks are related with fracture toughness, an attempt was made to correlate fracture toughness and (tensile index) x (stretch)$^{1/2}$. The correlation coefficient obtained was $R^2 = 0.76$, however a better correlation coefficient was obtained when stretch was replaced with TEA. As shown in Figure 15 the linear regression fitted to the data obtained from fracture toughness against (tensile index) x (TEA)$^{1/2}$ yielded the best correlation with $R^2 = 0.84$, compared to that of previously obtained values with single and combined tensile parameters.

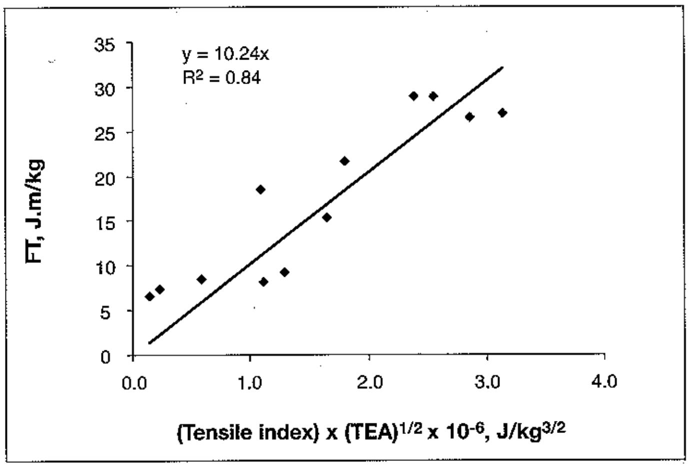


Fig. 15 Plot of fracture toughness against (tensile index) x (TEA)$^{1/2}$ x 10^{-6}.

Although there is a drop in the maximum error in the estimated fracture toughness values with combined parameters (78% with (tensile index) x (TEA)$^{1/2}$) the average errors (31%) are worse than that obtained from FT-tensile index.

As a final remark, these results indicate that the tensile properties of the samples tested can be used to obtain a general idea about the FT of these materials. However the relationships between tensile properties and FT are not strong enough to predict the FT of the material. This highlights the requirement for a method that can easily and rapidly evaluate the 'intrinsic property' of fracture toughness.

CONCLUSIONS

Correlations between tensile index-FT and TEA-FT with correlation coefficients of $R^2 = 0.72$ have been observed for the tested samples. The correlation coefficient was improved to a maximum of $R^2 = 0.84$ when tensile parameters were combined as (tensile index) x (TEA)$^{1/2}$. However, the relationships were not strong enough to justify using measurements of tensile properties to predict the fracture toughness. The FT data obtained for 'Reflex' copy paper suggest that the assumptions behind the EWF technique are not valid for longer ligament lengths.

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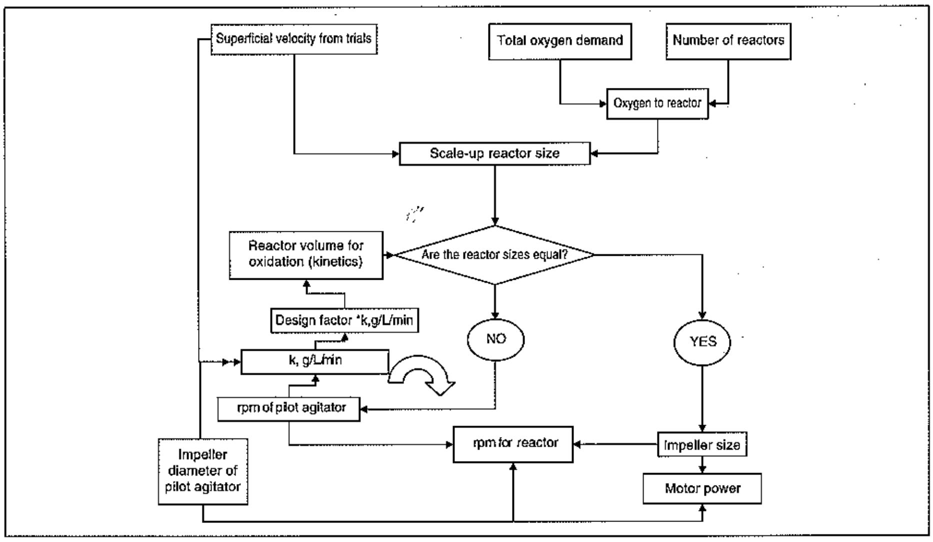

Fig. 7 **Scale-up procedure chart.**

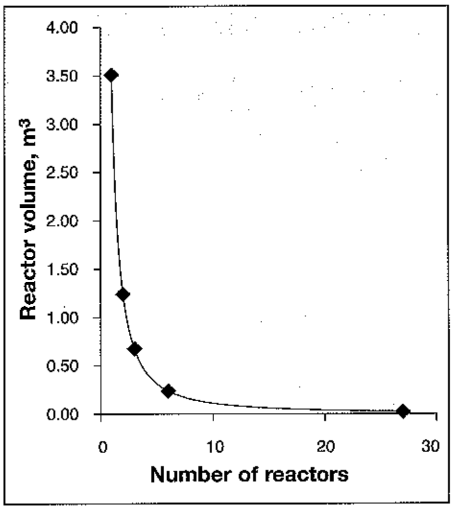

Fig. 8 **Effect of reactor number on the reactor volume – option 2.**

CONCLUSIONS

Green liquor oxidation is a zero order reaction under 'practical' mixing conditions. Under non-flooding conditions the oxidation reaction rate constant is strongly correlated with the oxygen superficial velocity and power/volume ratio. The reaction rate is temperature dependent, both kinetically and dynamically.

The GLOX plant capacity can be increased with the addition of CSTRs either before or after the existing reactor. For the same degree of GLOX plant expansion the later option will need less space-time and mixing power than the first option. When a greater number of reactors is utilised the total required space-time will be less but more mixing power will be needed.

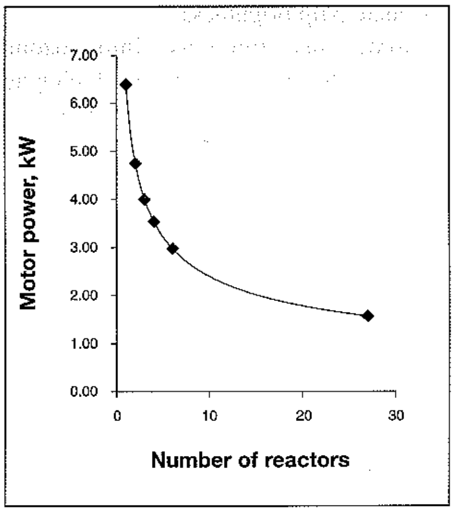

Fig. 9 **Effect of reactor number on the mixer motor size – option 2.**

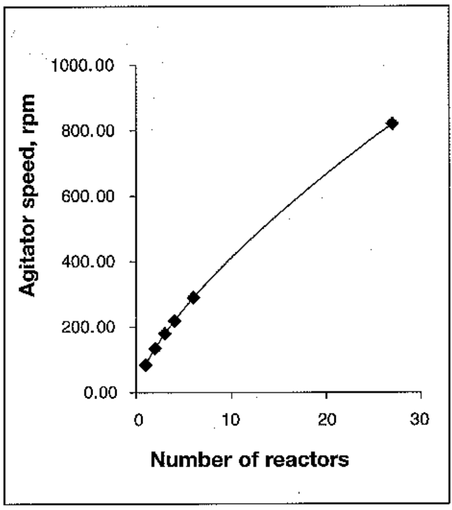

Fig. 10 **Effect of reactor number on the total mixing speed – option 2.**

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