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Measure of the internal bond strength of paper/board

Ahmed Koubaa and Zoltan Koran

ABSTRACT: Three methods are used to measure the internal bond strength of papers made from TMP, CTMP, and kraft pulps. These are the z-directional tensile test, the delamination test, and the Scott bond test. The comparisons between these methods using the same unit of measure (J/m²) show that even though the results are highly correlated, these tests do not measure the same thing. In addition to the fiber-to-fiber bond breakage energy, the delamination energy includes the energy dissipated in the fibrous network of thick papers, while the zdirectional tensile energy includes the energy of intrafiber bond failure especially for papers made from flexible chemical pulp fibers. On the other hand, the Scott bond energy is affected by basis weight and by the dynamic nature of the test which tends to over evaluate this parameter. This analysis further shows that the z-directional tensile strength is the best suited measure to determine the internal bond strength especially for high basis weight papers.

KEYWORDS: Basis weight, bibliographies, bonded area, bonding, bulk density, cohesion, delamination, fiber bonding, handsheets, measurement, paperboard tests, pulps, shear strength, tensile strength, test methods, z direction.

Legend

CBT: cantilever beam test DE: delamination energy DT: delamination test IBS: internal bond strength

PCS: peel cohesion strength

PCT: peel cohesion test RBA: relative bonded area S: light scattering coefficient S_o: light scattering coefficient for unbonded sheet

SBE: Scott bond energy SBS: specific bond strength SBT: Scott bond test

SCS: shear cohesion strength SCT: shear cohesion test ZDTT: z-directional tensile test ZDTE: z-directional tensile energy ZDTS: z-directional tensile strength

The internal bond strength (IBS) plays an important role in paper since poor bonding strength results in delamination and splitting in printing and coating operations. This property depends on the number of bonds, the average area per bond and their specific strength. It is also affected by both pulp properties and the treatments employed during the papermaking process. The internal bond strength can be measured on either single fiber crossings or on paper/boards. Several methods have been developed for each case. The results of these methods can be expressed in terms of strength, energy, or even as a modulus. These measures are generally highly correlated with one another, but they do not provide the real value of the fiberto-fiber bond strength. Therefore, the specific objective of this article is to compare three commonly employed methods using different kinds of pulps. These are the z-directional tensile test (ZDTT), the delamination test (DT), and the Scott bond test (SBT). It is hoped that these comparisons will help to provide a better fundamental understanding of the measure given by each method.

Internal Bond Strength

Materials

Various commercial and laboratory pulps (**Table I**) have been used to measure the IBS of paper. These pulps were beaten to various degrees in a PFI mill to ensure variation in fiber bonding.

Standard handsheets with a nominal basis weight of $60\,\mathrm{g/m^2}$ were made according to TAPPI Method T 205-om 88 from all pulps. In addition, handsheets were made at basis weights ranging from 15 to 200 $\mathrm{g/m^2}$ from a bleached softwood kraft (BSK) pulp.

Methods

Many methods have been developed to measure the IBS of paper/boards. These methods are based on tensile, shear or even dynamic loading and the IBS can be determined in terms of strength, energy or modulus.

Tensile methods

The peel cohesion test (PCT) is a tensile static test (Fig. 1A) that measures the resistance to splitting of paper/board when a force is applied at right angles to its plane (1-6). When carried on a free rotating wheel assembly (Fig. 1B), this test has been called the delamination test (DT) (2. *3).* In this method, the paper sample is sandwiched between two pieces of adhesive tape, the ends of which are pulled apart by hand until paper splitting is initiated. The force (N) required to complete fracture of the remainder length of sample (m) is then measured. The peel cohesion strength (PCS) can be expressed in N/m, in kPa, assuming that the sample has uniform thickness or even in energy per unit surface (J/m²).

In the present study, the delamination test (DT) was used to determine the IBS. The methods and preparation parameters to measure the delamination energy (DE) were the same as described previously (2).

The z-directional tensile test (ZDTT) is the oldest test used to

I. Pulp types and properties

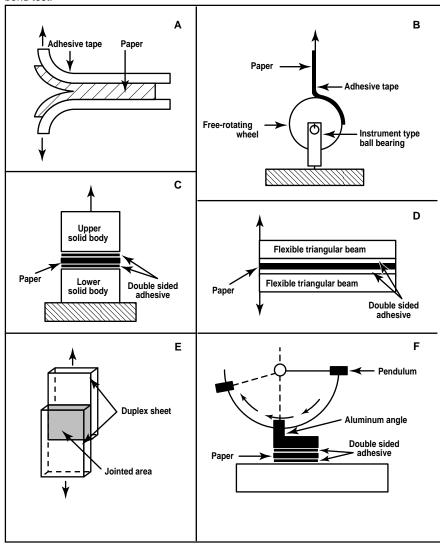
Type of pulp	Code	CSF variation, mL			
1 Stage softwood TMP	TMP	475, 295, 220, 130			
Bleached softwood kraft	BSK	675, 500, 380, 220			
Nonbleached softwood kraft	NBSK	625, 540, 380, 260			
Nonbleached hardwood kraft	NBHK	625, 530, 430			
Softwood CTMP	S CTMP 1	660, 530, 415, 310			
Softwood CTMP	S CTMP 2	540, 485, 400, 350			
Aspen CTMP	A CTMP	405, 375, 235, 125			
Birch CTMP	B CTMP 1	545, 415, 240, 160			
Birch CTMP	B CTMP 2	420, 335, 250, 200			

measure the IBS of paper/board (Fig. 1C). This is a static test that can be carried out according to TAPPI Method T 541 om-9 (7) and CPPA Standard Method D 37P (8) or by the use of a standard tensile tester (9, 10). In this test, a paper sample is sandwiched between two double sided adhesive tapes which, in turn, are glued to the two specimen holders to be inserted into testing equipment. The use of rigid adhesive such as epoxy instead of the double-sided adhesive was found to reinforce the IBS of paper (11) due to its penetration into the fiber network (12).

This test was used in this study to measure the IBS of paper. It was carried out on an Instron tensile tester at an extension rate of 20 mm/ min. The paper sample was sandwiched between two solid square bodies (645.16 mm²) by means of a pressure sensitive double-sided adhesive tape (3M No. 610). To ensure good adhesion of the double-sided adhesive to both paper and solid bodies, the samples have been pressed in a hydraulic press at 345 kPa for 1 min. These parameters have been checked and were found suitable for all pulps.

The z-directional tensile strength (ZDTS) is generally defined as the

1. Methods of measuring the internal bond strength: A. Peel cohesion test; B. Delamination test; C. Z-directional tensile test; D. Cantilever beam test; E. Shear cohesion test; F. Scott bond test.



force required to produce a unit area fracture perpendicular to the plane of paper (kPa). In the present study, a new parameter is introduced; this is the z-directional tensile rupture energy (ZDTE) and it is determined from the stress-deformation curve. The area under this curve represents this energy and is calculated according to Eq. 1:

$$ZDTE = \int_0^{\varepsilon} \sigma \ d\varepsilon \tag{1}$$

where

 σ = stress at failure, N/m²Pa

 ϵ = deformation at failure, m

ZDTE= z-directional tensile energy of rupture, J/m².

Schultz-Eklund *et al.* (13) measured the rupture energy of paper in the z-direction using the cantilever beam test (CBT). This is a tensile static test, where a paper sample is sandwiched between two triangular flexible beams with double sided adhesive (**Fig. 1D**). The force required to cause rupture and crack propagation along the paper sample is then measured. With the knowledge of the beam geometry and the bending stiffness, the energy per unit crack area (J/m²) is calculated and defined as the z-toughness.

Shear methods

The shear cohesion test (SCT) is generally used to measure the bonding ability of duplex sheets (1, 6). The shear cohesion strength (SCS) is obtained by measuring the shearing stress (kPa) applied in the plane of the sheet in order to separate the joint interface of a duplex sample (Fig. 1E). The main shortcoming of this method is the impossibility of the determination of the IBS single standard handsheets (60 g/m²). In fact, Byrd et al. (14) reported that paper samples must have a minimum thickness of 5 mm to determine their interlaminar shear properties.

The shear loading has been used in the determination of the bond strength of single fiber crossings (15–18). In theory, these are the only tests that give the real fiber-to-fiber bond strength. However, these tests need a great deal of skill in samples preparation, they are time consuming and are not reproducible since a very high variation has been reported (15, 16).

Dynamic methods

The Scott bond test (SBT) is a well known impact test employed for measuring the IBS of paper/board (19). The procedure involves laminating a test specimen between a metal block and one leg of a short length of aluminum angle with the aid of a doublesided adhesive tape (Fig. 1F). The block is placed on the frame of an instrument supporting a pendulum, which, upon release, strikes the upper edge of the angle, causing the sheet to split. A calibrated scale indicates the resistance to splitting in terms of the amount of the loss in potential energy during the swing of the pendulum. Therefore, the Scott bond rupture energy (SBE) is expressed in energy units for a unit surface produced in the test (J/m²). This test was employed in the present study.

The velocity viscosity product (VVP) test (20, 21) is another dynamic test that has the advantage of

II. Means and coefficients of variation (cv, %) of the ZDTS, ZDTE, DE and SBE for different pulps

Pulp	CSF, mL	ZDTS, kPa		ZDTE, J/m²		DE, J/m²		SBE, J/m²	
		Mean	cv, %	Mean	cv, %	Mean	cv, %	Mean	cv, %
TMP	475	124.1	5.5	24.4	7.9	91.25	1.8	16.3	11.2
BSK	380	697.0	1.7	339.6	5.3	66.7	0.9	356.5	12.2
NBSK	380	654.0	4.4	514.6	9.0	nd	nd	432.45	12.8
NBHK	430	730.4	7.8	268.1	6.2	66.81	0.4	227.9	12.2
SCTMP1	415	414.6	6.1	113.0	7.2	69.2	2.9	184.5	9.7
ACTMP	405	597.3	7.2	211.7	10.5	74.6	8.5	195.3	10.3
SCTMP2	400	416.5	6.8	90.0	7.4	56.7	7.0	118.3	12.2
B CTMP1	415	306.9	4.6	72.4	9.9	58.6	1.0	141.1	12.1
B CTMP 2	420	579.2	4.7	196.2	6.6	66.2	3.4	190.7	13

simulating the printing operation in a rotatory press. The device used in this test is made up of a pair of wheels. The paper sample is mounted on the lower wheel by means of double-sided adhesive tape. In this test, the upper wheel carrying a viscous film of polyisobutylene (PIB) of predetermined thickness and viscosity comes in contact with the lower wheel and applies a constant force to the specimen through the viscous film. During the test, both wheels turn in the same direction. The critical velocity for the known test film viscosity is determined for the onset and continuous propagation of fracture within the sheet. The IBS is determined by the viscosity velocity product and expressed in kp-cm/s units.

The measurement of the modulus of elasticity in the z-direction by ultrasonic wave propagation is another useful dynamic method to characterize the IBS (22, 23) and is expressed in Pa units. In addition to the nondestruction of the paper sample, this method is extremely fast and it does not require the use of external products such as adhesives. Furthermore, this method has the potential for on-line applications. Its main drawback is its low sensitivity

to the enhanced bonding by chemical strength aids (23).

Determination of the specific bond strength

The specific bond strength (SBS) and the cohesiveness are equivalent terms and are defined as the ratio between the IBS determined according to any of the above methods, and the net or actually jointed area (1, 24). The latter can be determined from the relative bonded area (RBA) or apparent density. The RBA is determined according to Eq. 2:

$$RBA = (S_0 - S)/S_0 \tag{2}$$

where S is the scattering coefficient of the sheet and S_0 is the light-scattering coefficient of the same sheet in the unbonded state. The determination of the last term can be obtained by the extrapolation of the S plot against the tensile strength (25), the modulus of elasticity (26) or even the z-directional tensile strength (9) to zero.

The change of the light-scattering coefficient during or after straining has been also used to determine the relative bonded area. The ratio between the straining tensile energy and the change in light-scattering

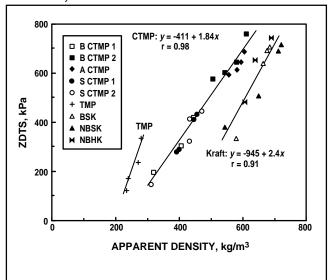
III. Specific bond strength (SBS) variation in different pulps

Code	SBS variation, kPa	
TMP	821-1886	
BSK	897-1591	
NBSK	1074-1530	
NBHK	1224-1655	
S CTMP 1	753-1480	
S CTMP 2	1114-1473	
A CTMP	1622-1742	
B CTMP 1	946-1471	
B CTMP 2	1716-1912	

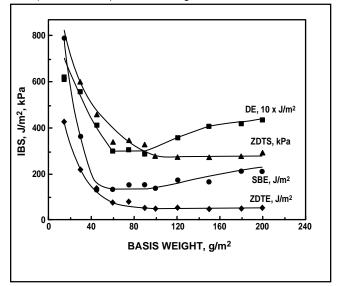
coefficient gives the Nordman bond strength (27). Skowronsky and Bichard (2) measured the increase in the light-scattering coefficient due to bond breakages after delamination and calculated the SBS.

Hieta *et al.* (28) used image analysis to measure the bonded area of fibers on cellophane after ZDTT rupture. The ratio between the ZDTS and the percentage of the paper-cellophane bonded area gives the bond

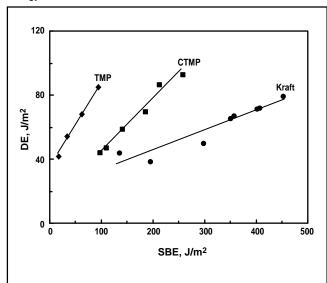
2. Variation of the z-directional tensile strength of various pulps with sheet density



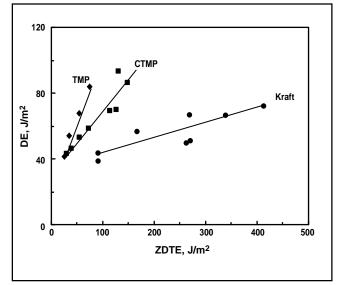
3. Variation of the internal bond strength of a bleached softwood kraft (CSF = 625 mL) with basis weight



4. Comparison of the Scott bond energy with the delamination energy



Comparison of the Scott bond energy with the z-directional tensile energy



strength index. The latter is equivalent to the SBS.

The optical determination of the RBA to calculate the SBS is possible in the case of chemical pulps while in the case of high yield pulps, this is impossible since light scatters through the lumen of fibers. In this case, the density data could be used to determine the SBS since density and the RBA are very close. In fact, Hieta et al. (28) reported a linear relationship between the apparent

density of various chemical pulps and the percentage of the bonded area determined by image analysis (conformability index). Furthermore, Stratton (23) showed that the extrapolation of the linear relationship between the apparent density and light-scattering coefficient (S) to zero for classified chemical pulps falls at $1620~{\rm kg/m^3}$. This is very close to the density of fiber wall ($1530~{\rm kg/m^3}$). If we assume that the latter corresponds to a completely bonded

sheet, it is possible to determine the SBS of the various pulps using apparent density data according to Eq. 3:

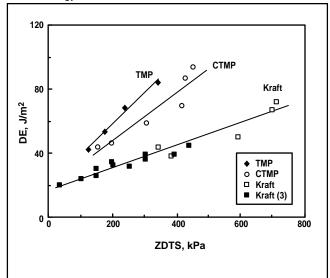
$$SBS = IBS \times 1530/\rho \tag{3}$$

where ρ is the sheet apparent density.

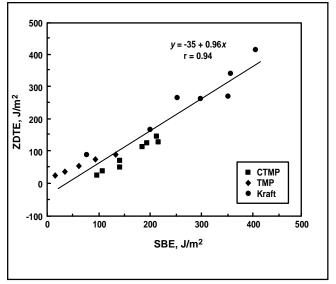
Results

The IBS of paper was evaluated by four different parameters including

6. Comparison of the delamination energy with the z-directional tensile energy



7. Comparison of the Scott bond energy with the z-directional tensile strength



the ZDTS, ZDTE, DE, and SBE. Means and coefficients of variation of these parameters at comparable CSF are presented in **Table II**. **Figures 2 and 3** show the effects on the IBS of sheet apparent density and basis weight, respectively. **Table III** presents the range of variation of the SBS as determined from ZDTS and density data for all pulps according to Eq. 3.

Furthermore, comparisons were made between the three methods in terms of energy (J/m²) and strength (kPa) units. These comparisons are illustrated in **Figs. 4 through 8**. The use of the same unit of measure in these comparisons is necessary to have better fundamental understanding of the measure given by each method. This is illustrated in Figs. 4, 5, and 7 using the energy unit as a basis of comparison. On the other hand, Figs. 6 and 8 compare the ZDTS to the DE and the SBE, respectively.

Discussions

An increase in paper density as a result of beating or higher compacting pressures during wet pressing is universally observed to correspond to an increase in fiber-to-fiber bond-

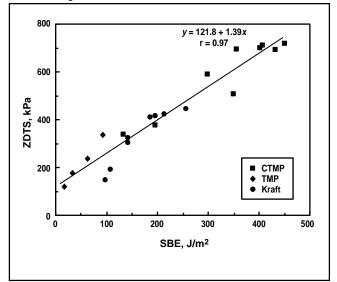
ing (6, 9–11, 23, 29). In the present study, increase in density has been achieved by beating and its effect on the ZDTS is shown in Fig. 2. As expected, ZDTS increases linearly with apparent sheet density. However, this relationship differs for TMP, CTMP and kraft pulps.

It is apparent that the kraft pulps yield the highest densities and the TMP the lowest, while the CTMP fall in between. These differences are due to the variation in fiber flexibility. In fact, it is well known that the kraft pulp fibers are more flexible than the CTMP fibers, which in turn are more flexible than the TMP fibers. However, in terms of ZDTS, Fig. 2 shows that the TMP sheet vields the same ZDTS (325 kPa) at low density (280 kg/m³) as the CTMP (400 kg/m³) or the kraft pulp at high density (530 kg/m³). This is due to the fact that the refining process in high-yield pulping produces important changes in the fiber surface producing fibrillation which enhances the SBS. The data of Table III and the use of Eq. 3 support this affirmation. In fact, at 325 kPa ZDTS, the TMP has the highest SBS (1775 kPa), and the kraft pulp the lowest (940 kPa); the CTMP falling in between (1243 kPa). For the kraft pulps, the SBS found in this study (Table III) are of the same order of magnitude as those reported by Hieta *et al.* (28).

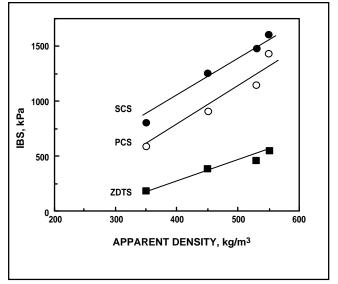
It is interesting to note that the different types of CTMP yield a linear relationship (Fig. 2) between the ZDTS and the apparent density (y = -411 + 1.84x) with a very high coefficient of correlation (r = 0.98)and an acceptable coefficient of variation (12.5%). This indicates the close relationship between these two properties and the possibility of predicting the ZDTS from the density data. However, this prediction is possible only in the absence of chemical aids and under identical wet pressing conditions. In fact, it has been reported that sheet density is not very sensitive to chemical strength aids, while the ZDTS is affected significantly by the same parameter (23, 30). It was also noted that the ZDTS/density relationship depends on whether densification has been achieved via wet pressing or refining (23).

The same holds for the kraft pulps where a similar relationship (Fig. 2) was found (y = -945 + 2.4x) with a high coefficient of correlation (r = 0.91). It is interesting to note that hardwood and softwood data fall on the same curves for both CTMP and

8. Comparison of the delamination energy with the z-directional tensile strength.



9. Comparison of the shear cohesion strength and of the peel cohesion strength with the z-directional tensile strength (6).



kraft pulps (Fig. 2). This suggests that fiber morphology does not affect these relationships. However, in both cases, hardwoods yield higher sheet densities and ZDTS than softwoods. This can be attributed to the shorter fibers in hardwoods which tend to form denser sheets of paper and paperboard.

Intercomparison of the various methods

The coefficient of variation (CV) data (Table II) provide an important statistical tool to compare the precision of the various measures. It is evident that the SBE produces the highest CV (to 12.8%), followed by ZDTE (to 10.5%), ZDTS (to 6.8%), and by DE which showed the lowest value (to 5.3%). These data show that DE and ZDTS parameters offer the highest degrees of precision and reproducibility.

The variation of IBS with basis weight offers a powerful tool to compare the relative value of each one of these measures. Although similar curves are shown (Fig. 3), there are important differences between them.

In every case, the high IBS at low basis weight is due to the reinforcing effect of the adhesive. The increase in basis weight produces significant decreases in both the ZDTS and ZDTE followed by a leveling-off from about 100 to 200 g/m². This can be explained by the analogy that the weakest link in a chain is the one which determines its overall strength. The longer is the chain, the greater is the probability of having a weak link in it. By the same analogy, the probability of having weak zones in the z-direction increases with increase in basis weight or in sheet thickness. Furthermore, the leveling-off suggests that, in multi-layered sheets made from the same pulp, the weakest zones have almost the same strength.

In the case of ZDTE, the leveling-off is surprising. In fact, when the basis weight increases, it is supposed that the overall deformation in the z-direction increases and so does the energy which is not the case (Fig. 3). The occurrence of deformation in the zone of rupture only is a plausible explanation.

In contrast to the ZDTS and ZDTE curves, an increase in basis weight results in an increase in DE. This was explained by the dissipation of energy in the fibrous network as the sheet changes from a mono- to a multi-layered structure (3). In fact, the failure mode in the peel tests depends on the peel angle and sample thickness (31). At a 90° peel angle and thin papers, it is assumed that only tensile failure occurs (23). However, in the case of thick samples and at peel angles different from 90°, an in-plane shear component contributes to the failure, thus resulting in higher rupture energies.

In the case of the SBE curve (Fig. 3), the increase in failure energy as a function of basis weight can be explained by the increased number of partial breakages in the thicker sheet, which consume energy without causing full failure.

All of the above observations suggest that basis weight is an important factor in the determination of the IBS of paper/boards. Figure 3 shows that in the case of SBT and DT, the IBS should be measured on thin papers (60 g/m²) to avoid an over evaluation of this parameter, while the ZDTT provides constant values at 100 g/m² basis weight and higher. Therefore, the ZDTT is most suitable for paper/boards and multilayered sheets.

Figure 3 further shows that at the same basis weight, SBE is consistently higher than ZDTE. This has been explained by the dynamic

Internal Bond Strength

nature of the SBT and by the possible occurrence of rupture in the fiber wall (3). The ZDTE is in turn higher than DE because of the energy consumed in the breakage of intrafiber bonds. Another comparison is made between DE and SBE in Fig. 4 for TMP, CTMP, and kraft pulps. It is apparent that the SBE values are generally higher than those of DE. This is due to the dynamic nature of the SBT and to the possible occurrence of rupture in the fiber wall (3). Figure 4 further shows that relationships between the SBE and the DE differ from one pulp to another. The variation in the slope with the pulp type shows that these two measurements are affected differently. In fact, it is well known that kraft fibers have better bonding abilities than the CTMP and TMP fibers, respectively. This is confirmed by the SBT which shows that the kraft pulps yield the highest SBE and the TMP the lowest, while the CTMP are in between. In contrast, the DT does not confirm this since the TMP showed very high DE for low SBE comparatively to the CTMP and krafts. At 100 J/m² SBE, for example, the DE values are 82, 40, and 30 J/m², respectively, for TMP, CTMP, and kraft pulp. The high DE values of TMP can be attributed to the thicker sheet at the same basis weight in comparison with CTMP and kraft pulps, respectively. With analogy to Fig. 3, the energy dissipated in the TMP fibrous network, due to the action of a shear component, is no longer negligible. The same holds for the CTMP that shows higher DE than the kraft pulps.

A similar family of lines is obtained in Fig. 5 when ZDTE is plotted against DE. Even the slopes of the three lines seem similar to those in Fig. 4, and the comparison yields similar conclusions. At the same ZDTE (90 J/m²), the TMP yields higher DE (93 J/m²) than the CTMP (65 J/m²) and kraft pulps (39 J/m²), respectively. These variations can be explained once again by the fact that standard handsheets (60 g/m²) made

from TMP are thicker than those from CTMP or from kraft pulps.

Figure 5 further shows that at the same DE significantly different ZDTE values are obtained for the three types of pulps. For example, 70 J/m² DE corresponds to 50, 95, and 340 J/m² ZDTE values, respectively, for the TMP, CTMP and kraft pulps. These differences can be explained by the fact that the ZDTE includes both intra- and inter-fiber bonding rupture energies. In fact, it is well known that the TMP fibers are rigid, and they keep their cylindrical shape after wet pressing and drying. Consequently, the energy of the intrafiber bonding rupture is nil. In contrast, the kraft pulp fibers are very flexible elements; consequently, they are completely collapsed after wet pressing and drying. When strained in the z-direction, these fibers tend to open up and the energy consumed for intrafiber rupture is very high. The flexibility of CTMP fibers occupy an intermediate position in between; they are partially collapsed after wet pressing and drying. Consequently, when strained in the z-direction, there is a certain amount of energy consumed to break intrafiber bonding.

Since the ZDTE includes the energy of intrafiber bonding, it is interesting to compare the DE to the ZDTS. This comparison is shown in Fig. 6, where a family of similar lines is obtained. This enhances the conclusions drawn from Figs. 4 and 5 which stated that, in the case of TMP and CTMP, the DE includes an amount of energy dissipated in the fibrous network. It is also interesting to note that Stratton's kraft data fall on the same line as the experimental data (Fig. 6). In contrast to Figs. 4, 5, and 6, single linear relationships are obtained for the three different kinds of pulps when ZDTE and ZDTS are plotted against SBE (Figs. 7 and 8). The high coefficients of correlation suggest that both ZDTE and ZDTS are very close to SBE. However, it should be noted that the SBE is consistently higher (35 J/m 2) than ZDTE (Fig. 7). This is due to the dynamic nature of the SBT and to the possible occurrence of rupture in fiber wall (3).

The ZDTS has been compared (6) to the peel cohesion strength (PCS) and the shear cohesion strength (SCS) for duplex sheets made from chemical pulps (Fig. 9). It is evident that at any given density, the SCS values are higher than those of PCS. This is due to the fact that the force applied in the SCT is divided into two components: the force required to shear the joint, and the force acting in the plane of the sheet. On the other hand, the PCS values are higher than those of ZDTS. This is due to the possible action of a shear component in the thickness of the duplex sheet.

All of the above comparisons suggest that all energy per unit surface measurements include the energy consumed in fiber-to-fiber failure and other energies. The ZDTE, for instance, includes both intra- and inter-fiber bonding rupture energies; this is especially evident from kraft pulps data. On the other hand, DE includes the energy dissipated in the fibrous network especially in the case of thick papers. Similarly, SBE is over-evaluated because of the dynamic nature of the SBT and the possible occurrence of rupture in the fiber wall.

The ZDTS is highly correlated with DE, SBE, PCS, and SCS and is affected by neither sample thickness nor pulp type. Consequently, in order to avoid an over-evaluation of the IBS by various energies and shear components, the ZDTS is the most suitable measure of the IBS especially for multi-layered sheets and paperboards. Furthermore, this measure is precise and reproducible (Table II).

Conclusions

Chemical pulps have better bonding abilities than CTMP and TMP fibers. However, the latter have the highest specific bond strength. Even though the IBS provided by three energy methods are highly correlated, they do not measure the same thing. In fact, each measure is affected differently. The SBE is overevaluated because of the dynamic nature of the SBT, the ZDTE includes the intrafiber bonding energy, while the DE includes the energy dissipated in the fiber network for thick papers. In order to avoid this over-evaluation of the IBS, the ZDTS seems to be the most suitable in the determination of IBS for all kinds of pulps.

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