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THE EFFECT OF SAMPLE SIZE AND SHAPE ON THE HYGROEXPANSION COEFFICIENT - A STUDY MADE WITH ADVANCED METHODS FOR HYGROEXPANSION MEASUREMENT.

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Hygroexpansion, Dimensional stability, Optidim, laser measurement, Eucalypt pulp

ABSTRACT

The hygroexpansivity of industrial hardwood pulp samples dried in restraint was determined by using two principally different kinds of measurement apparatus: A laser measurement for paper strips and a method based on the digital correlation technique (OPTIDIM) using square-shaped samples. The purpose of this investigation was to study the differences in calculated hygroexpansivity values between these measurement methods and to investigate whether this was a result of the measurement method used, or a result of some other phenomenon involved with the measurements.

The results differed greatly, while the ranking of pulp samples in terms of their hygroexpansivity was roughly the same. The hygroexpansivity measured with the laser measurement technique was in most cases 20% higher than that obtained using digital correlation. The shape and size of the samples were not enough to explain the differences. Therefore the differences observed between the paper strips and square-shaped samples were probably due to the initial stress of the clamping device holding the paper strips, or the local variations in thickness, fibre orientation and fibre grammage.

The differences were lowest for unrefined fibres. The poor correlation between the measurements methods seems to indicate that the differences arise from the measurement procedure itself. Direct comparisons between the hygroexpansion values measured by these two kinds of apparatus should therefore be avoided.

INTRODUCTION

Hygroexpansion is a paper property of great interest in many applications, for instance in printing operations, but also in relationship to the mechano-sorptive creep. Paper expands or shrinks as the relative humidity in the local environment changes. When hygroexpansivity is plotted against relative humidity, paper generally shows complex hygroexpansion behaviour, dependent on its humidity history before testing and during testing (Uesaka, 1994a,b; Salmén, 1993).

Various types of apparatus have been developed to measure the hygroexpansivity of paper. Neenah-type hygroexpansimeters have been used in the industry for years (Poustis and Vidal 1994; Van den Akker et al. 1942). The basic design of the Neenah-type hygroexpansimeter has been further developed in various research institutions by replacing the micrometer with a displacement or position sensor to detect the hygroexpansion. (Ikuta et al. 1996; Serra-Tosio 1994; Uesaka 1992). In this study, an improved version of the STFI apparatus was used (based on laser measurement of the length change of paper strips) altered at STFI Packforsk.

Digital correlation techniques have also been applied to hygroexpansion measurements (Kajanto 1996; Lif et al. 1995). A speckle pattern is created on a paper surface, either by spraying colored particles or by using natural speckles (by utilizing surface roughness or basis weight variation). Images are taken by a high-resolution charge-coupled device (CCD) camera and then digitized. These techniques make it possible to obtain detailed information on two-dimensional hygrodeformation without physical contact. One disadvantage is that out-of-plane deformations such as curl and cockle (Glynn and Jones 1959; Kajanto 1992) that occur during the humidity change significantly deteriorate the accuracy of this technique.

In this study, both normal handsheets (isotropic)

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and dynamic sheets (anisotropic) were studied by using two principally different measurement methods to investigate how sensitive the measurement procedure of hygroexpansivity in reality is and to ponder whether the results are comparable or not. The effect of the size and shape of the sample was then studied with the OPTIDIM apparatus.

EXPERIMENTAL

Materials

Bleached industrial hardwood kraft pulps (*Eucalyptus grandis/E. dunnii*, Uruguay; *E. globulus*, Portugal; *E. urograndis*, Brazil; *Betula pendula/B. pubescens*, Finland; *Acacia magnium*, Indonesia; *E. urograndis/E. grandis*, South Africa; *E. grandis /E. saligna*, Brazil) were obtained from pulp mills as dry sheets. The second part of the study, the effect of size and shape of the samples, was made utilizing only E.grandis/E.dunnii.

Refining

Unbeaten pulps as well as pulps beaten with 50 kWh/t and 150 kWh/t in a Voith Sulzer laboratory refiner, using an edge load of 0.4 J/m. The consistency of the pulp suspension was 4.0 % and 2/3-1.46 40D fillings were used.

Sheet making and paper testing

Laboratory sheets with a grammage of 60 g/m^2 were prepared in a conventional sheet former according to ISO 5269-1:1998. Anisotropic sheets were made with a dynamic sheet former (Uesaka, 2002) at the KCL research centre. The wire speed was set to 1000 m/min. The jet speed was set to 920 m/min (1.6 bar) and the feed concentration was 4 g/l. The grammage of dynamic sheets was 65 g/m^2. Wet sheets were cut and then pressed according to the standard. ISO 5269-1:1998. Both the isotropic sheets and the anisotropic sheets were dried in contact with a plate to which the sheets were attached. This affected shrinkage during drying, which was done in a climate-controlled room (23 °C and a relative humidity of 50%). Both, the samples studied with the OPTIDIM and the laser measurement device (STFI packforsk, Stockholm, Sweden) were prepared in a similar fashion at the KCL research centre. Grammage and thickness were determined according to ISO 536:1995 and ISO 534:2005, respectively, except that the apparent thickness was measured on four sheets for all pulps. Tensile properties were measured according to ISO 5270:1998.

Carbohydrate measurements

Carbohydrate composition of the pulps was determined using the following method; utilizing sulphuric acid hydrolyzes at 120°C to degrade the carbohydrates into monosugars, based on Tappi standard T222-om0. Monosugars were then analyzed by ion chromatography (Metrohm 817 Bioscan system).

OPTIDIM

OPTIDIM is an optical method for measuring the dimensional stability of paper under varying humidity conditions. The sample, which was placed in a humidity control cabinet and after each humidity change and after equilibrium had been reached with the surrounding air moisture content and the moisture content of the sample, was put between weighted glass plates to prevent any out-of-plane dimensional changes and transilluminated with a bright light from the bottom and a picture was taken from above with a CCD camera (Figure 1). By placing the weight on top of the sample just before a picture is taken, will minimize the friction between the glass and the sample. The glass weight was chosen according to ISO 8226-2:1990 based on the grammage of the handsheets. The variations in brightness level caused by the formation of the paper were measured. The dimensions of the samples at low relative humidity were compared with the dimensions at higher relative humidity. The dimensional changes as a function of relative humidity were then calculated. (Kajanto et al. 1996).

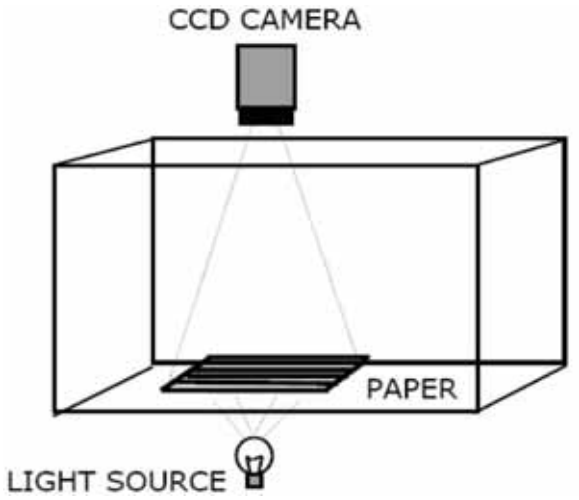


Figure 1. The measurement principle of the OPTIDIM measurement apparatus.

Laser measurement device

Hygroexpansion was measured on test pieces subjected to a relative humidity of 66%RH prior to testing. The measurements were thereafter made on an instrument developed at STFI Packforsk, similar to a previous version described by Salmén (1993). It consists of 30 rigid clamps and 30 freely movable clamps, with 100-mm gaps between the clamps in which 30 paper strips are placed independently in a horizontal position. A weight is placed on the strips to eliminate any effects of buckling on length registration. These clamps were placed in a chamber with regulated humidity. The relative humidity was changed from 50±2%RH to 22±3%RH to 33±2%RH to 66±2%RH and the differences in lengths were measured between 33±2%RH and

66±2%RH in accordance with ISO 8226-1:1994. The apparatus is described schematically in Figure 2.

The weights used on top of the strips can be seen in Figure 3 and are placed upon the strips at the length registration marks to eliminate any effects of buckling. However, these weights will not place a load on the paper strips and hence no mechanical stretch is applied. Clamps are placed in a chamber into which air with regulated humidity streams. The movements of the movable clamps are measured by laser reading and two reference points are used for calibration.

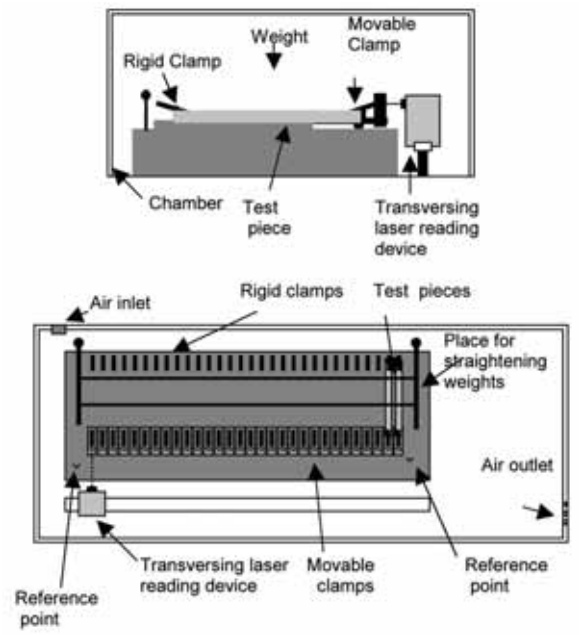
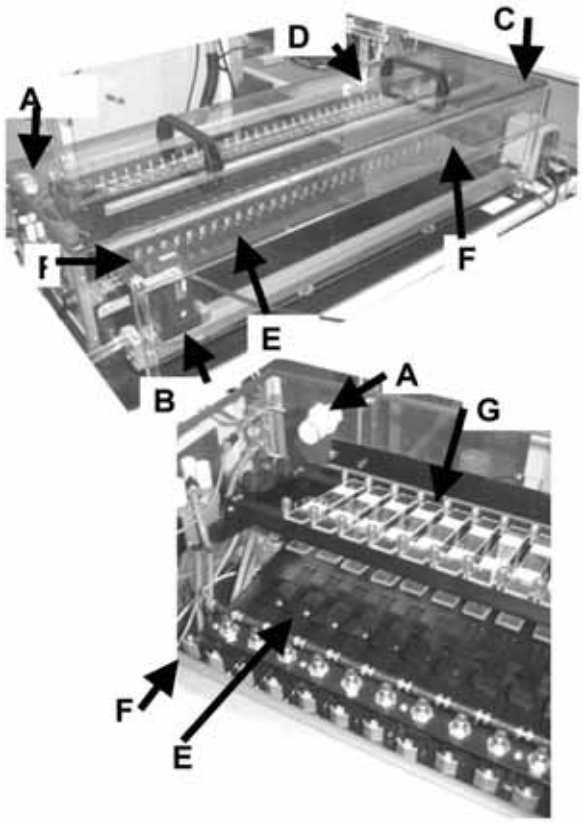


Figure 2: Schematic description of the design of the laser measurement equipment (STFI Packforsk).



Measurement procedure with OPTIDIM
Six parallel square-shaped sheet samples (7 x 7 cm) were prepared for each sample in each of the refining points of both conventional handsheets and dynamic sheets. The confidence intervals for OPTIDIM measurements of hygroexpansivity coefficient (Nanri 1993) were roughly 0,01 for isotropic sheets and 0,007 for dynamic sheets (confidence level of 95%). With OPTIDIM, both directions, machine and cross (MD and CD), are measured simultaneously. Relative humidity was changed linearly from standard (50% RH) to low (10% RH), then back to 50% RH and to higher humidities (70% RH and 90% RH) and back to standard (50% RH) humidity for 4 h per cycle from one relative humidity to another at 23 °C. The samples were also weighed at the moisture content used and thus the moisture content (%) of the paper can be measured. The hygroexpansion coefficient (β) was determined from the gradient of the linear part of the curves in the low-moisture range and is expressed as hygroexpansional strain divided by moisture change (Nanri 1993).

β= dimensional change (%)/MC change (%) (1)

Samples of different size and shape
The samples were taken from the dynamic sheets cut both in the machine and the cross direction. The samples sizes used in this study were 100x100 mm², 60x100 mm², 36x100 mm² and 15x100 mm², so the actual number of samples was eight. The normal procedure for hygroexpansion measurements was applied to all the samples with the exception of the 15x100 mm² samples, when the use of the glass weight was excluded. This may have resulted in curling, making it difficult to get accurate readings of sample expansion. Therefore a large number of parallel measurements were required and the results for 15x100 mm² remain highly unreliable.

Measurement procedure with the laser device
Paper strips with a width of 15mm were made from conventional sheets as well as oriented sheets both in the MD and the CD. These strips were subjected to a relative humidity of 66%RH prior to testing. They were then placed between the clamps, divided by a distance of 100 mm, in the hygroexpansion measuring device and the humidity was changed from 50±2%RH, to 22±3%RH, to 33±2%RH, to 66±2%RH and back to 50±2%RH in accordance with ISO 8226-1:1994. The difference in length of the strips was measured every 15 minutes and after 6 hours, at 33%RH and 66%RH respectively, the paper strips were considered to be in equilibrium with the air humidity and the value of the length difference at these points was used to determine the difference in length. The confidence intervals for the hygroexpansion coefficient measurements for

Figure 3: Pictures of the laser measurement device. A: Air inlet; B: Laser reading device; C: Air outlet; D: sensor for registration of %RH and temperature; E: Movable clamps; F: Reference points; G: Straightening weights.

laser measurement device were somewhat smaller than those of OPTIDIM. To obtain a hygroexpansion coefficient based on the moisture content in the sheets, $\beta_{\%MC}$, the moisture content of the pulp strips was determined separately by placing the strips in the different humidities for a period of time long enough to achieve a state of equilibrium and to determine the content of moisture gravimetrically. The bone dry weights were determined after the strips were placed in an oven at 105°C for a minimum of 12 hours.

RESULTS

Figure 4 shows the hygroexpansion curves obtained from both of the hygroexpansivity meters used in this study. With the method used to calculate the hygroexpansion coefficients (1), the values obtained are both in the range of linear deformation of the sample. The values are therefore comparable. Figure 5 shows the correlation between all hygroexpansivity measurements of the OPTIDIM and the laser measurement device (presented in Table 1). There seems to be a poor correlation between these two measurements, even though the magnitude of the results is approximately the same. The forming of hydrogen bonds at a low moisture range (Urquhart and Eckersall, 1930; Urquhart, 1958) may affect the results. The preconditioning of the samples differed somewhat. In the measurements with the strips, the samples were preconditioned at 33% RH, whereas the OPTIDIM samples were kept at +23ºC and 50% RH prior to testing. It is a common practise to condition the paper first at a substantially lower humidity (e.g. 30% or lower) before it is tested in the standard conditions in order to achieve identical moisture content at a given relative humidity (Wink, 1961). As shown in Figure 4, there is no hysteresis behaviour with the laser measurement device due to the lower RH range used.

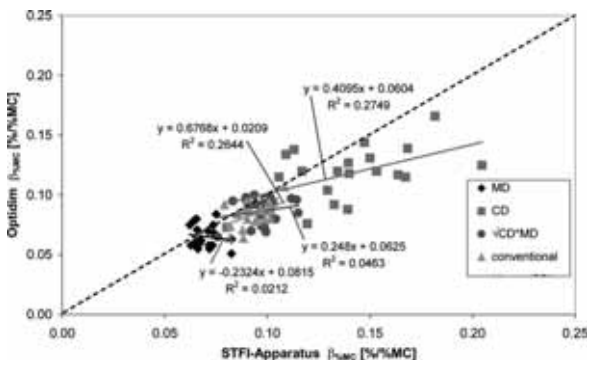


Figure 5: All data points from conventional sheets as well as the MD and CD of oriented sheets.

		OPTIDIM			Laser measurement device		
		Refining energy (kWh/t)					
	Sample	0	50	150	0	50	150
		Hygroexpansivity coefficient (%/%)					
Isotropic handsheets	*E.grandis/E.dunnii*	0.09	0.07	0.09	0.10	0.09	0.08
	E.globulus	0.06	0.09	0.09	0.09	0.09	0.10
	E.urograndis	0.10	0.08	0.08	0.09	0.10	0.10
	B.pendula	0.07	0.08	0.09	0.08	0.09	0.10
	Acacia magnium	0.08	0.09	0.11	0.10	0.10	0.11
	E.urograndis/E.grandis	0.09	0.08	0.09	0.09	0.09	0.10
	E.grandis/E.saligna	0.06	0.08	0.08	0.09	0.09	0.09
Anisotropic handsheets							
- Geometric mean	*E.grandis/E.dunnii*	0.10	0.09	0.08	0.08	0.10	0.10
	E.globulus	0.07	0.08	0.09	0.10	0.10	0.12
	E.urograndis	0.07	0.07	0.09	0.10	0.10	0.10
	B.pendula	0.10	0.10	0.10	0.09	0.10	0.12
	Acacia magnium	0.10	0.10	0.10	0.09	0.10	0.11
	E.urograndis/E.grandis	0.10	0.09	0.09	0.09	0.09	0.10
	E.grandis/E.saligna	0.07	0.07	0.09	0.08	0.09	0.10
- MD	*E.grandis/E.dunnii*	0.08	0.07	0.06	0.07	0.07	0.07
	E.globulus	0.06	0.06	0.06	0.07	0.07	0.07
	E.urograndis	0.06	0.06	0.06	0.07	0.07	0.08
	B.pendula	0.07	0.06	0.06	0.07	0.07	0.07
	Acacia magnium	0.08	0.08	0.07	0.07	0.07	0.07
	E.urograndis/E.grandis	0.07	0.08	0.08	0.07	0.08	0.08
	E.grandis/E.saligna	0.06	0.05	0.06	0.06	0.08	0.07
- CD	*E.grandis/E.dunnii*	0.12	0.12	0.12	0.10	0.13	0.18
	E.globulus	0.09	0.12	0.13	0.14	0.14	0.20
	E.urograndis	0.08	0.09	0.12	0.12	0.14	0.15
	B.pendula	0.14	0.14	0.17	0.11	0.14	0.18
	Acacia magnium	0.12	0.13	0.14	0.11	0.14	0.17
	E.urograndis/E.grandis	0.13	0.10	0.12	0.11	0.13	0.17
	E.grandis/E.saligna	0.07	0.09	0.13	0.08	0.11	0.14

Table 1. Results of the hygroexpansivity measurements for all samples.

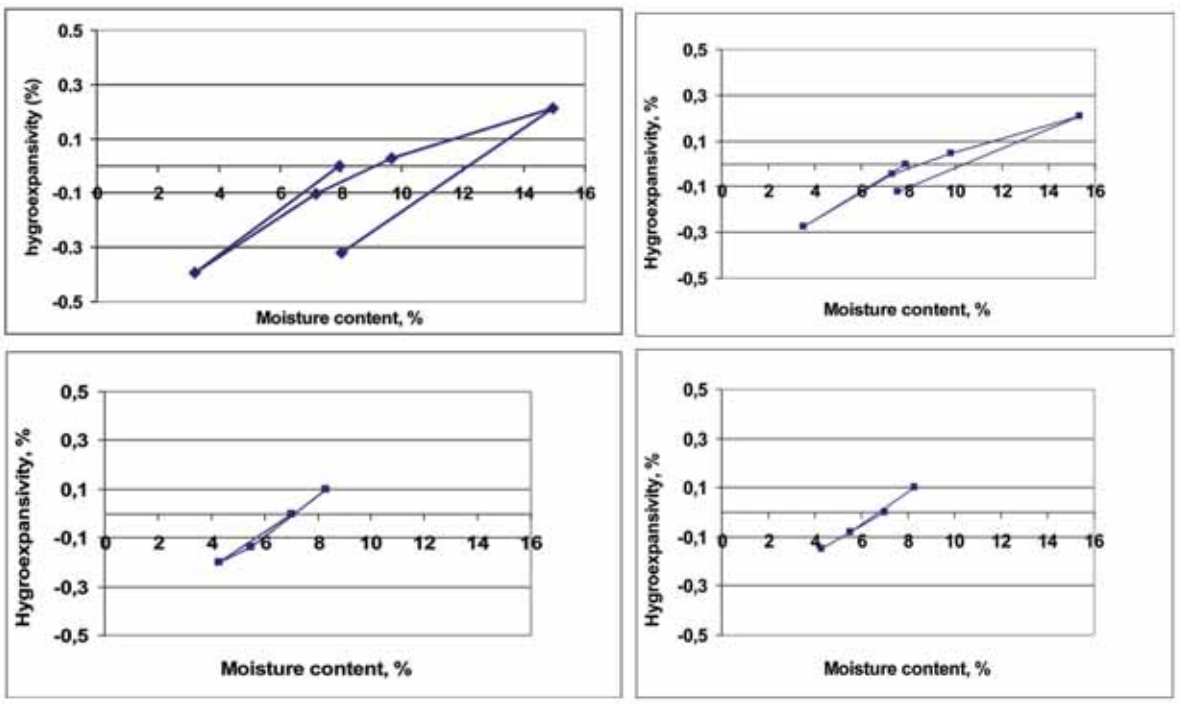


Figure 4: Hysteresis curves of the OPTIDIM (above) and the laser measurement samples (bottom) for oriented sheets in the CD (left) and in the MD (right).

Another difference that could have affected the results is the low humidity end of OPTIDIM (10% RH). When the relative humidity is less than 20%, the samples undergo a steep change in moisture content with increasing relative humidity (Salmén, 1993). The OPTIDIM samples at high relative humidity (between 70% and 90% relative humidity and even before that) exhibit non-linear behaviour due to the release of "internal stresses" (Uesaka et al. 1989; Uesaka et al. 1992; Salmén 1993). However, this did not affect the results as the hygroexpansion coefficient was calculated from the linear part of the curve. The moisture cycles used in this study were the most commonly used moisture cycles for each measurement device. The OPTIDIM apparatus could be adjusted to use a cycle between 33% RH and 66% RH, but as it would require a lot of additional work and, in authors' opinion, would yield no significant change in the results, the normal cycle of between 10% RH and 90% RH was used.

There were no significant correlations between the samples at similar refining levels (table 1). Also, the coefficients of hygroexpansion for isotropic handsheets as a function of refining were non-existent. The generally accepted fact that the hygroexpansivity as a function of refining in the MD is quite unpredictable and can even decrease in some occasions and that the hygroexpansivity in the CD generally increases with increased refining (Salmén et al. 1987; Uesaka and Qi 1994b) was observed for both methods of this study. The hygroexpansivities of all of the samples were roughly of the same order of magnitude, including eucalypt, acacia and birch. No indication of the lower hygroexpansivity of acacia was observed, as stated earlier by Liu and Retulainen (2003). Figure 6 shows the correlation between samples of conventional sheets for both measurement apparatuses for all points measured. Even though made with the same Scandinavian sheet former, the sheet making process is very delicate and small differences in formation can occur due to fibre deformations (curl etc.).

In order to compare the results of isotropic handsheets and anisotropic sheets there needs to be approximately similar fibre orientation between each measurement group (i.e. isotropic sheets and anisotropic sheets in the MD and CD). Table 2 lists the MD/CD tensile stiffness index ratios used to characterize the anisotropy of the handsheets. If the orientation of the fibres and fibre curl is to be constant for all eucalyptus and acacia samples, when examining the fibre network we can see that the hygroexpansion is controlled by the transverse shrinkage of fibres (Uesaka 1994a, b). The birch sample differs in orientation (controlled as MD/CD tensile stiffness index- ratio) for the un-refined sheet. The effect of fibre wall thickness values and the effect of refining on hygroexpansion has been discussed earlier by Pulkkinen et al. 2009. The geometric mean values measured for both apparatuses are presented in Figure 7.

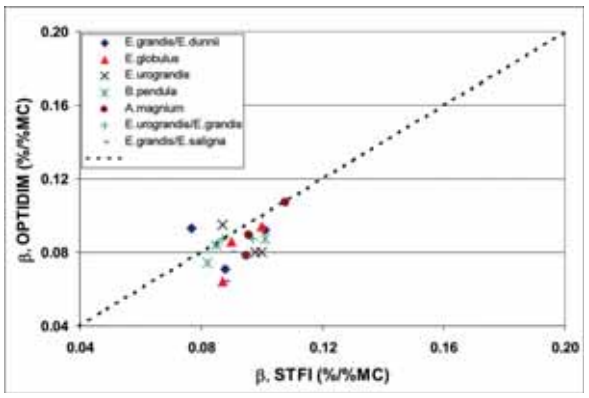


Figure 6: The comparison between the OPTIDIM and the laser measurement device results for conventional sheets.

Sample	Refining (kWh/t)	MD/CD $_{TSI}$	Curl (%)
E.grandis/E.dunnii	0	2.47	20.9
	50	2.48	20.3
	150	2.02	21.4
E.globulus	0	2.33	18.2
	50	2.72	17.5
	150	1.68	19.7
E.urograndis	0	2.28	18.2
	50	2.34	17.5
	150	1.97	19.6
B.pendula	0	1.42	21.1
	50	3.03	20.1
	150	2.34	21.6
Acacia magnium	0	2.35	22.2
	50	2.36	21.7
	150	1.91	21.2
E.urograndis/E.grandis	0	2.32	18.2
	50	2.42	17.4
	150	2.07	18.5
E.grandis/E.saligna	0	2.47	18.8
	50	2.59	17.1
	150	2.29	18.3

Table 2. MD/CDTSI ratios and fibre curl as a function of refining

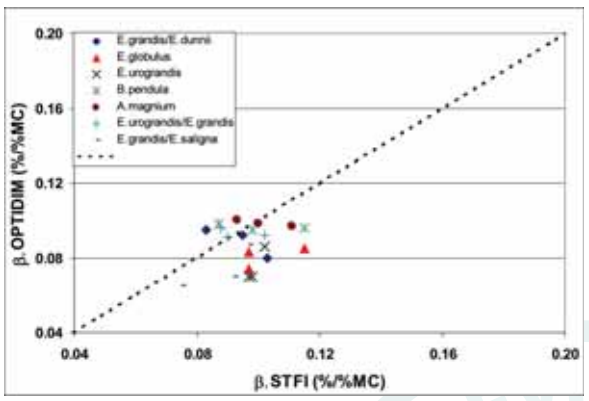


Figure 7: The correlation between the OPTIDIM and the laser measurements for the geometrical mean value of the dynamic sheets.

Figures 8 and 9 depict the hygroexpansion coefficients of isotropic handsheets as a function of the geometric

mean values of machine direction and cross direction hygroexpansion coefficients for laser measurement device and OPTIDIM hygroexpansivity tester, respectively. Interestingly, some of the measurements made with the laser device (Figure 8) fall on a straight line. For example, all the measurement points of acacia and E.urograndis follow the line nicely.

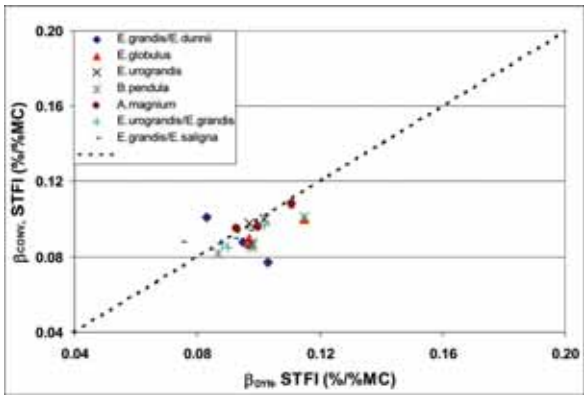


Figure 8: Comparison of the hygroexpansion of conventional and dynamic sheets with the laser measurent device.

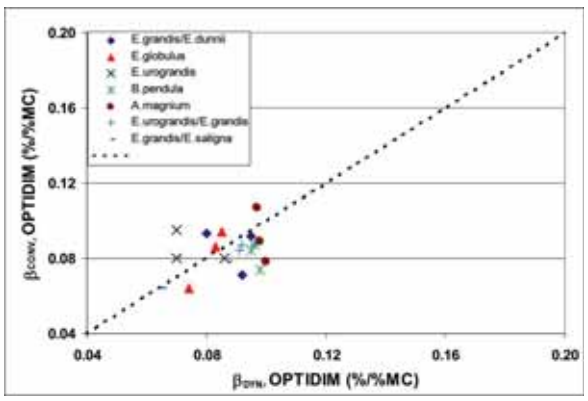


Figure 9: Comparison of the hygroexpansion of conventional and dynamic sheets with OPTIDIM.

There is no theoretical basis that these values should correlate. The measurements (made for sheets dried in restraint), as stated before, are mostly governed by the transverse hygroexpansion of the fibres. In the case of isotropic handsheets, where fibres are placed randomly to form a sheet, the hygroexpansional forces are distributed evenly in all directions therefore activating the network equally.

For anisotropic sheets, depending on the amount of anisotropy, fibre network activation in the machine direction and cross direction is dependent on the morphological properties of the fibres (Uesaka 1994a; Uesaka and Moss 1997)). As the fibre segment activation is affected by the free segment length and fibre curl and the length of the free segment in the MD and CD is affected by the fibre orientation, the hygroexpansion in the MD can behave quite unpredictably. This is due to the amount of fibres aligned in the MD and CD. The more fibres aligned in the MD determine how many bonds there are in the CD (Lyne, 1994). Relatively straight fibres together with high swelling ability at the crossing sites would mean that refining has little or no effect on the hygroexpansivity of the sheets in the MD. The amount of activation depending on the shrinking ability of the fibres at the fibre bonding sites means that the results for isotropic sheets and anisotropic sheets are not necessarily comparable with each other.

Table 3 shows that the difference in the shape or size of the sample was not reflected in the hygroexpansivity of the samples when using the OPTIDIM measurement apparatus. Increasing the moisture content of the sample will change the internal stress state of a fibre and, because of the Poisson effect (Perkins 2002), the moisture-induced stresses observed in one direction will have an effect on the stresses in the other direction. Therefore, in theory, with changing aspect ratios of the MD and CD side lengths, one should see differences in these values. Judging by the high values of hygroexpansion in the CD measured with the laser device (Table 1), one could speculate that the ratio of MD and CD lengths is meaningful and the OPTIDIM measurement device was unable to measure the dimension changes properly.

This difference in the values observed was especially seen with the refined fibres. The refining alters the bonding potential and internal and external structure of the fibres, i.e. increasing the number fibre-to-fibre interaction. Due to the increased bonded area the paper produced is less dimensionally stable. The initial creep effect, possibly induced by the clamping of the samples in the laser measurement apparatus, could also be intensified when the samples from refined fibres were prepared, as refining lowers the stiffness of the fibres.

	100mm x 15mm	100mm x 36mm	100mm x 60mm	100mm x 100mm
Longer side cutted in MD				
β_{MD}	0.01	0.06	0.06	0.06
β_{CD}	0.07	0.08	0.08	0.08
Longer side cutted in CD				
β_{MD}	0.05	0.07	0.06	0.06
β_{CD}	0.08	0.09	0.08	0.09

Table 3: The effect of shape and size of the sample on the hygroexpansivity in the MD and CD.

Small differences can be seen with the samples of paper strips. For such a sample, local variations in thickness, fibre orientation and fibre grammage may become important. Such variation may result in heterogeneous sheet hygroexpansion, causing inaccuracy during the measurement procedure when going down in sample size.

The percentual differences between the laser measurements and the OPTIDIM tester were approximately 20% for most of the measurements. This leads to the speculation mentioned above, whether initial creep is involved in the measurements using the laser measurement apparatus due to the

clamps used to hold the samples. The differences in the measurement accuracy were too small to explain the differences observed. The possibility exists that there could also be a significant friction force between the glass and the sample in the OPTIDIM apparatus or between the weight and the sample in the laser measurement device. Batchelor and Westerlind (2001) used Teflon sheets to allow free shrinkage of the sample when determining stress-strain curves.

A similar experimental set-up could be advantageous in determining the hygroexpansivity coefficients in stress-free conditions for a set of pulps. Teflon-coated glass plates could be useful for dimensional change measurement with both types of apparatus.

CONCLUSIONS

In this study, two advanced methods to measure the hygroexpansion of paper samples were studied; one utilizing a digital correlation technique with square-shaped samples and the other using laser measurement to detect the length change of the paper strip. There were differences in the level of the hygroexpansivity coefficients calculated from the results of the apparatuses studied; the results between the apparatuses did not correlate either. It is strongly recommended to avoid a direct comparison between these two methods. However, a few possible reasons emerged to explain these differences. The use of paper strips may at least be partially responsible for the differences observed.

For such a sample, local variations in thickness, fibre orientation and fibre grammage may become important. Another possible cause was the clamps used with the laser measurements to place the samples in the humidity chamber. This may have resulted in a small initial creep that can accelerate the relaxation of the dried-in stresses in the samples.

In addition, there is also the possibility of the increased importance of the creep behaviour when sheet bonding is affected negatively. This could explain the significant differences in the hygroexpansivity values found in the case of birch samples.

Overall, there are many things that have to be taken into consideration when measuring the hygroexpansion of samples using different kinds of apparatuses, especially when dealing with samples that are relatively small in size (standard strip or smaller). It is impossible to create identical circumstances, but further studies could be executed with identical relative humidity cycles to remove uncertainties concerning either the low humidity range, or the high humidity range and the use of Teflon coated weights could also be considered.

ACKNOWLEDGEMENTS

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