

# A microscopic study of fibre-fibre contacts in paper

JIHONG HE\*, WARREN J. BATCHELOR† AND ROBERT E. JOHNSTON‡

## SUMMARY

Confocal microscopy is used to acquire images of paper cross-sections in which some longitudinally sectioned fibres are chosen as fibres of interest. The properties of fibre-fibre contacts including the number of fibre-fibre contacts per unit length of fibre, the nature of the contacts, the free fibre lengths and the out-of-plane deflection angles of the free fibre segments with respect to those fibres of interest are determined directly in these images. It is found that both the shape of fibre cross-section and the length of fibre have no significant effects on the properties of fibre-fibre contacts. The measured distributions of free fibre lengths cannot be fitted to the negative exponential distribution proposed by Kallmes and Bernier. However a two-parameter Weibull probability density function seems to provide an acceptable fit to the measured distributions. It is demonstrated that model structures of fibre-fibre contacts can be reconstructed using the measured data.

## Keywords

Confocal laser scanning microscopy, number of fibre-fibre contacts, free fibre length, out-of-plane deflection angle, free fibre length distribution, fractionation, fibre cutting, pressing

Properties of fibre-fibre contacts in paper, including the number of fibre-fibre contacts per unit length of fibre, the nature of contacts, the free fibre length and its distribution, and the out-of-plane deflection angle of a fibre segment, are the most important fundamental properties of a paper structure. Most previous studies of fibre-fibre contacts focused on the development of models for number of fibre-fibre contacts using statistical methods either based on analysis of model

structures of paper (1,2) or fibre assemblies (3-5). These models have provided valuable knowledge for understanding the fibre-fibre contacts in paper. However, verification of these models is always problematic because there is little experimental data on the properties of fibre-fibre contacts in paper. There is always a requirement for new techniques for measuring comprehensive data of fibre-fibre contacts directly in a sheet.

Some techniques have been used for measuring fibre-fibre contacts directly in a sheet, including examining the paper plane (x-y plane) of very thin fibre networks by their magnified projected images (1), examining the x-y plane of a normal sheet under vertical illumination in polarised light (2,6), and examining serial cross-sections of paper embedded in resin (7). The technique of Page et al. (6) and the technique of Kallmes et al. (2) can only measure fibres on the paper surface, and can only measure interbond distances between bonds on one side of the fibre. Page et al. (6) estimated the interbond distances within the sheet by superimposing two sets of measurements, one set representing the upper surface of the fibre and the other the lower surface. Yang et al. (7) examined a series of images of paper cross-section acquired at different depths from the paper cross-sectional surface, and measured the probability of fibre-fibre contacts with a given fibre cross-section. This technique overcomes the limitations of only measuring the surface fibres, however, it cannot measure the interbond distances. In a later study, a new technique was proposed to evaluate the 3-dimensional geometric arrangement of fibres in a paper sheet (8). Using this technique, the authors measured the possibility of fibre-fibre contacts with respect to a given fibre cross-section and the number of fibre-fibre contacts per unit length of fibre. The sample size examined by this technique was quite small (0.2 x 0.2 mm), therefore may not be fully representative. It appears that a more powerful technique is required for measurement of fibre-fibre contacts directly in a paper sheet.

Different parameters have been used to represent a free fibre length. The free fibre length has been defined as the distance between the centres of fibre crossings (1); the intercrossing distance represented by the distance between the centres of bonded crossings (9), and the projected interbond distance, which is the distance between the projection of the bonds on the fibre axis (6). Kallmes and Bernier (9) have shown that these three definitions of a free fibre length will have different absolute values but will produce the same distribution of the free fibre length. In this study we define the free fibre length as the distance between the centres of two neighbouring contacts or bonds, which is the same as that used in (9).

The aim of this study is to examine the fibre-fibre contacts directly in handsheets made from different furnishes using a combined technique of resin embedding and confocal microscopy. The effects of fibre transverse dimensions and fibre length on fibre-fibre contacts are investigated. The effects of wet pressing on fibre-fibre contacts are also examined. Model structures of fibre-fibre contacts are reconstructed using the measured data.

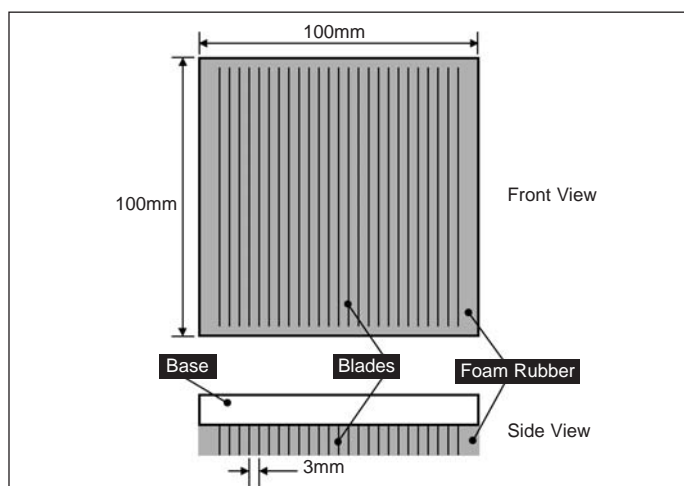
## MATERIALS AND METHODS

Laboratory made never-dried radiata pine kraft pulp, cooked to 45.6% yield with Kappa number 30.0, was used in this study. A Kajaani FS200 was used to measure all average fibre lengths and length distributions, which were reported in this study.

### Generating fractions of fibres with different transverse dimensions

The pulp fibres were fractionated with an AKW (Amberger Kaolinwerke GmbH) 40 mm cyclone in order to obtain fractions of fibres with different transverse dimensions. The fractionation was performed at 0.1% pulp stock concentration, and the accepts to rejects ratio was set to 90%. The separated accepts and rejects fractions were collected by draining the water and fines through a 200-mesh screen.

\* PhD Student,  
 † Senior Lecturer, Corresponding author,  
 ‡ Professor  
 Australia Pulp and Paper Institute,  
 Department of Chemical Engineering,  
 Monash University,  
 Victoria 3800, Australia.



**Fig. 1** Diagram of the die used to cut wet handsheets (drawing not to scale).

### Generating fractions of fibres with different fibre length

Wet handsheets were cut, immediately after they were formed on a Moving Belt Sheet Former (MBSF), using a specifically designed die (see Fig. 1) to generate fractions of fibres differing only in fibre length. The blades of the die were very sharp and thin so that the compression effects on the fibres during cutting was minimised. In the cutting operation, the die was placed blade side down (Front view) on several wet sheets and impact pressed. Three fractions of fibres with different fibre length were then created as

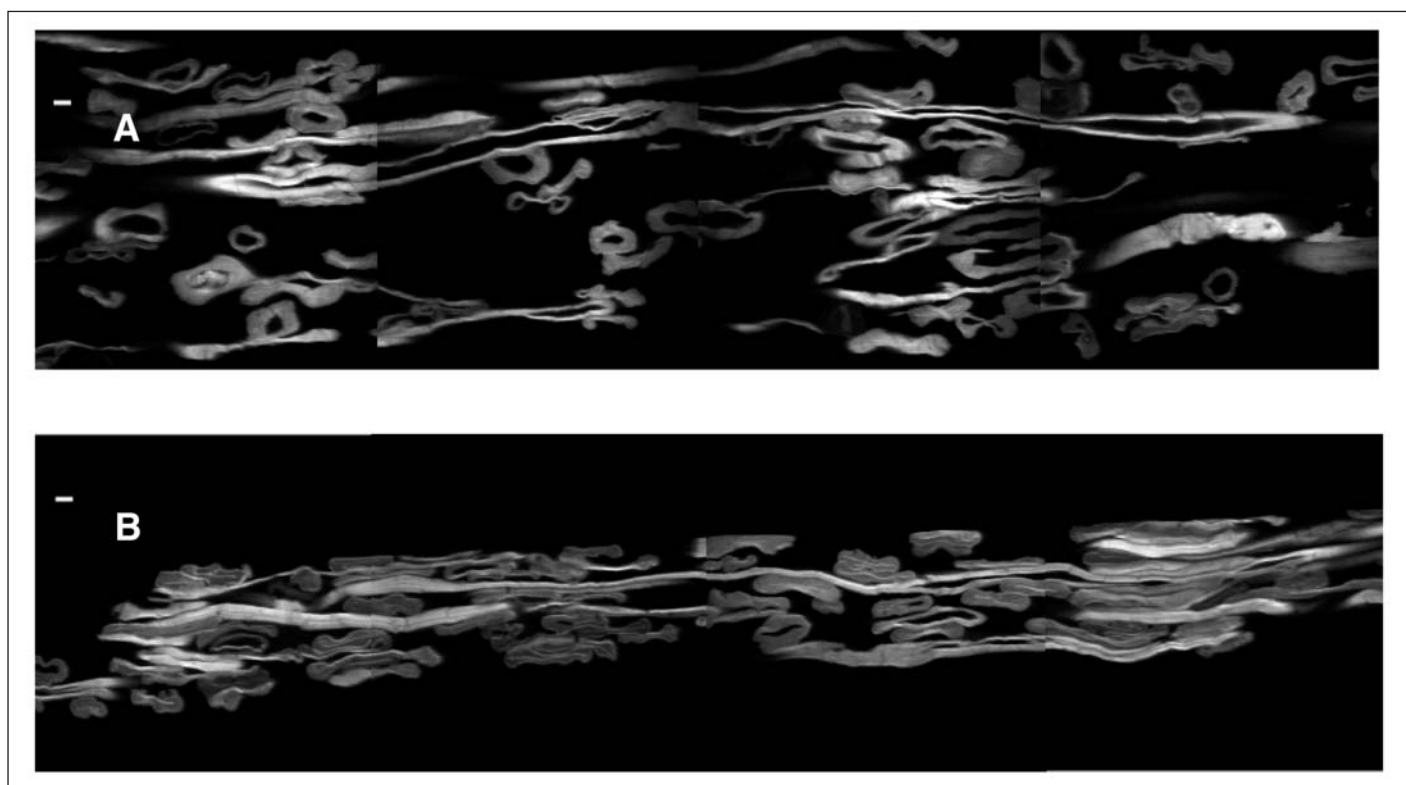
$L_0$ : uncut fibres,  $L_1$ : cut once in one blade direction,  $L_2$ : cut twice, with the die being rotated  $90^\circ$ . The fibre length of the three fractions was measured using a Kajaani optical fibre length analyser.

### Making handsheets from different fractions of fibres

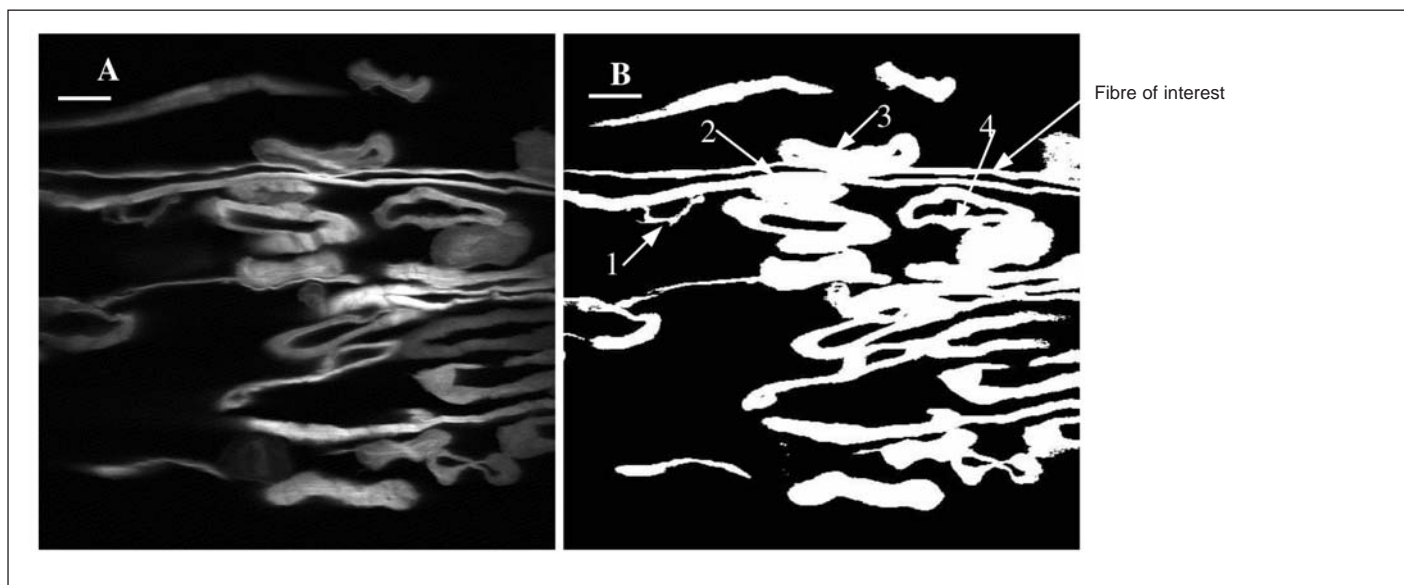
Handsheets from different fractions of fibres generated by fractionation and cutting wet handsheets were made on a Moving Belt Sheet Former (10). All of the fractions of fibres were dyed with Acridine Orange prior to handsheet formation.

Three sets of handsheets were made from the three fractions with different fibre lengths respectively. These handsheets were pressed statically using a hydraulic pressing machine at a pressure of 500 kPa. Each handsheet, sandwiched between blotter paper and an iron plate, was pressed for 2 minutes so that the handsheet was transferred to the iron plate. Each set of handsheets was denoted by 'S' plus the notation of the corresponding length fraction used, viz..  $SL_0$ ,  $SL_1$  and  $SL_2$ .

For the fractions of fibres generated by the fractionation, viz.. the accepts and the rejects, three sets of  $60\text{g/m}^2$  handsheets were made from the accepts, and three sets were made from the rejects. Each set of handsheets made from each of the fractions was pressed dynamically using a Sheet Roll Press at one of three pressing levels, viz. pressed one pass without applying additional load (denoted as  $P_0$ ), pressed one pass at  $7\text{ kN/m}$  (denoted as  $P_M$ ), and pressed one pass at  $3\text{ kN/m}$ , one pass at  $10\text{ kN/m}$  and ten passes at  $20\text{ kN/m}$  (denoted as  $P_H$ ). Each handsheet was sandwiched between an iron plate and blotter paper, the sandwich was placed between two press felts, which were then passed through the Sheet Roller Press. Each set of handsheets was denoted by using the pulp name and then the pressing level. For example,  $AcP_0$



**Fig. 2** Images of paper cross-section (A is sample Rej-P0 and B is Rej-PH).  
\* The size bar is  $10\ \mu\text{m}$



**Fig. 3** Cross-section image before (A) and after (B) thresholding and binarisation. Fibre 2 and 3 in (B) make two full contacts, fibre 1 makes a partial contact, and fibre 4 is not in contact with the fibre of interest.  
\* The size bar is 20  $\mu\text{m}$

represents handsheets made from the accepts and pressed at pressing level  $P_0$ , while  $RjP_M$  represents handsheets from the rejects and pressed at pressing level  $P_M$ .

All of the handsheets used in our experiments were dried under restraint at 23°C and 50% rh.

### Microscopic measurement of fibre-fibre contacts

The measured parameters associated with the fibre-fibre contacts included the nature of fibre contact, the number of fibre-fibre contacts, the length of free fibre segment and the out-of-plane angle of each free fibre segment. These parameters were measured directly in images of paper cross-sections. For each sample, 70 frames of cross-sectional images were obtained consecutively along the sample cross-section using a confocal laser-scanning microscope and a 60x oil-immersion lens. The inspection area of each image was 200x200  $\mu\text{m}^2$ . The 70 images were then joined together in consecutive order during the measurements; therefore a 14mm length of the sample cross-section was imaged in this way. Some fibres or fibre segments in the sample cross-section were imaged along their long axes. These fibres were chosen as fibres of interest for the measurements of fibre-fibre contacts. Two images of such fibres are shown in Figure 2. When such a fibre was chosen, the number of fibres in contact with the fibre of interest was counted, and a line was drawn between the centres of two neighbouring contacts along the fibre

axis. The length of the line is the free fibre length and the angle between the line and the horizontal frame of the image is the measured out-of-plane angle of the free fibre segment. If the horizontal frame of the image is at an angle  $-\alpha$  to the paper plane, then it can be shown that, for a randomly oriented handsheet, the average value of the out-of-plane angles of all the free fibre segments in the paper is equal to  $\alpha$ . We estimated the angle  $\alpha$  by averaging the measured out-of-plane angles and we then corrected all the measured out-of-plane angles by the angle  $\alpha$ .

To identify fibre-fibre contacts, the cross-sectional images of the samples were first thresholded manually, and then binaried into black and white images. In the binary images, fibre-fibre contacts could easily be identified. If there is no black gap between two fibre surfaces, we defined that as a contact that had been made between these two fibres. Otherwise, there was no contact between them. In this study, fibre-fibre contacts were classified into full contacts and partial contacts. The cross-section of a fibre is assumed to have two long sides and two short sides. When one of the long sides is totally in contact with the fibre of interest, this contact is a full contact (see Fig. 3). Otherwise, the contact is a partial contact (see Fig. 3).

The method of sample preparation for the confocal microscope involves embedding pre-cut samples in an epoxy resin and grinding the resin block to expose the cross-sections of the samples with pro-

gressively finer abrasive paper. Details of the method are described in (11).

## RESULTS AND DISCUSSION

An apparent density factor, AD factor, was defined in a previous study (12) as the ratio of the fibre wall area to the lumen area of a fibre. This AD factor was used to evaluate the fibre separation occurring in the hydrocyclone. The AD factors of the accepts and rejects were determined from measurement of the perimeter and wall area of fibres deposited on glass slides using the confocal microscope following the method described in (12). Figure 4 shows the cumulative distributions of the AD factors of the accepts and rejects fractions. Clearly there is a difference between the accepts and the rejects, with the rejects fraction having a higher average AD factor than the accepts. This indicates that the pulp fibres have been partially separated on the basis of different cross-sectional shape by the hydrocyclone operation.

Figure 5 shows the length-weighted fibre length distributions of the fibre fractions generated by cutting the wet handsheets. This method has been used in previous studies (13,14) and is believed to be able to change only fibre length while keeping other fibre dimensions constant. As shown in Figure 5, the length-weighted fibre length distribution is shifted to a lower value range and narrowed after the fibres were cut by the above operation. The average length-weighted fibre

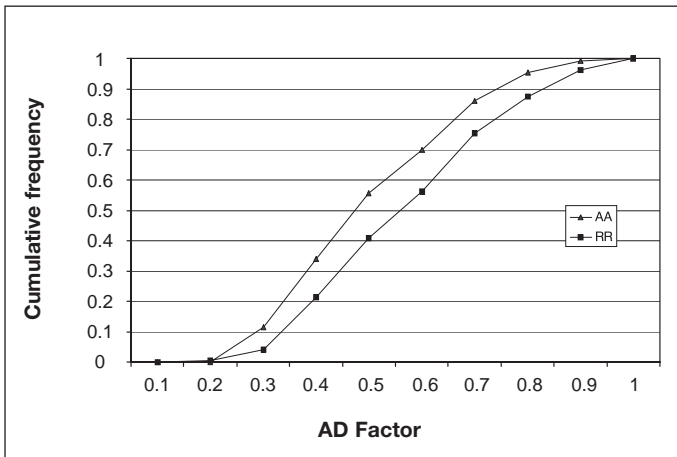


Fig. 4 Cumulative distributions of AD factor of the accepts and the rejects.

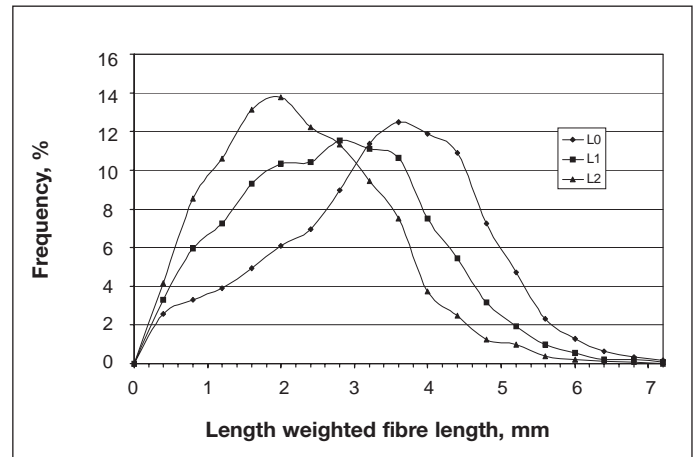


Fig. 5 Fibre length distribution of the fractions generated by cutting wet handsheets.

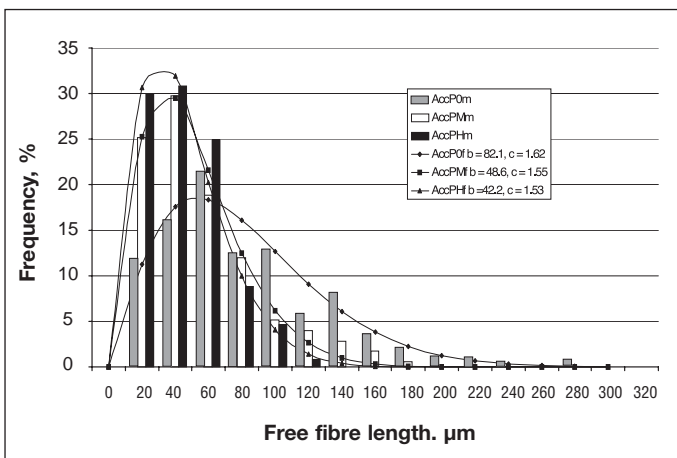


Fig. 6 Frequency distribution of free fibre length of samples of the accepts.

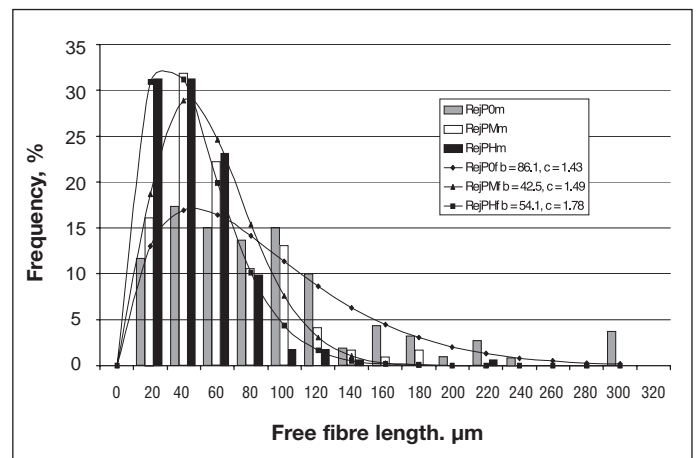


Fig. 7 Frequency distribution of free fibre length of samples of the rejects.

lengths of  $L_0$ ,  $L_1$ , and  $L_2$  were 3.14 mm, 2.53 mm and 2.10 mm respectively. These results show that cutting the wet handsheets had successfully reduced fibre length.

The distributions of free fibre length of the samples made from the accepts and the rejects are given in Figures 6 and 7 respectively. At a given pressing level, the free fibre length of the accepts sample shows a very similar distribution to that of the corresponding sample of the rejects fraction. Further examination of the average results (Table 1) shows that, at the same pressing level, the sample of the accepts and the sample of the rejects have almost the same values of the average free fibre length and number of fibre-fibre contacts per unit fibre length. As discussed above, the accepts and the rejects have different fibre cross-sectional dimensions (different AD factor). However, the two sets of handsheets made from these pulps have almost the same properties of fibre-fibre contacts, for a given level of wet pressing. It seems that for the sheets made from these pulps,

the cross-sectional dimensions of the fibres have no effect on the frequency distribution of the free fibre length and the number of fibre-fibre contacts per unit fibre length. Kallmes and Bernier (9) have shown that the free fibre length distribution is independent of fibre width, which is in agreement with the results obtained in this study. Page et al. (6) claimed that the free fibre length and the frequency of bonding are highly sensitive to the fibre width and to the fibre flexibility, but no particular experimental data were provided to support these claims. These results conflict with the results acquired in this study. One reason is that the definition used in this study, which completely ignores the influence of fibre width on the free fibre length, is different from the definition used by Page et al. (6). We believe that the fibre width and fibre flexibility mainly affects the bonded area of each bond. It is necessary to perform further experiments to draw a final conclusion.

The free fibre length distributions were skewed to a lower value range and nar-

rowed when the samples were pressed (see Fig. 6 and 7), resulting in decreases in the mean value of free fibre length and increases in the number of fibre-fibre contacts per unit length of fibre (Table 1). The response to wet pressing was the same for both the accepts and the rejects. The broad distributions of free fibre length in  $AccP_0$  and  $RejP_0$  imply that the bonding structure along fibres in these sheets is non-uniform, which will produce a non-uniform load distribution along the fibres and will lead to less efficient load transfer between fibres when the sheet is loaded. Wet pressing can narrow the distribution of free fibre length in the sheet and therefore improve the uniformity of load transfer between fibres when the sheet is loaded.

As shown in Table 1, sample  $AccP_0$  has higher percentage of full contacts than sample  $RejP_0$ . After wet pressing, full contacts in samples from both fractions increase dramatically, indicating, in another aspect, the increase in the degree of fibre bonding. We should also note that

each full contact in accept samples has higher contact area, on average, than that of a full contact in reject samples.

Page (6) believed that fibre length would have a strong influence on the frequency of fibre bonding, but no evidence was given. Kallmes and Bernier's (1) theory was based on fibres with infinite length, but their theory was partially verified with data obtained with normal sheets. In this study the effects of fibre length on the fibre-fibre contacts were also examined. Cutting wet handsheets, as discussed before, isolated the effects of fibre length on fibre-fibre contacts. As shown in Figure 8, the distributions of the free fibre length of these samples made from fibres with different fibre length are almost the same. It was also found that the mean values of the free fibre length, as well as the number of fibre contacts per unit length of fibre, are also the same at a 95% confidence. The results suggest that fibre length has no effect on fibre-fibre contacts, which conflicts with Page's findings (6). This raises an interesting point that would be worth further examination in the future.

In this study we used a two-parameter Weibull probability density function (PDF), as shown by Equation 1, to describe the distribution of the free fibre length.

$$f(g) = \frac{c}{b}(g/b)^{c-1} \exp(-(g/b)^c) \quad [1]$$

where  $b$  and  $c$  are constants, and  $b, c > 0$ , and  $g \geq 0$ .

The Weibull parameter  $b$  is the scale parameter, and  $c$  is the shape parameter. The values of  $b$  and  $c$  for different samples were estimated by non-linear least-squares curve fitting. The fitted lines are shown in Figures 6-8, and the values of  $b$

and  $c$  for the lines of best fit are given in these figures as well. The values of  $b$  are about 10  $\mu\text{m}$  higher than the corresponding mean of the free fibre length (Table 1). Different fit lines with different values of  $c$  for sample AccP<sub>M</sub> are shown in Figure 9. As can be seen in Figure 9, when  $c$  is equal to 1, the Weibull PDF becomes a negative exponential distribution and does not fit the experimental results well. Kallmes and Bernier (1,9) proposed that the free fibre length is given by the negative exponential distribution  $f(g) = (1/\bar{g})e^{g/\bar{g}}$ , where  $g$  is the free fibre length and  $\bar{g}$  is the mean of the free fibre length. They found this equation to hold for 2-dimensional sheets which are almost completely bonded (9). However this equation clearly does not hold for the data presented in this paper. In fact, the distributions of free fibre length measured in this study, as shown in Figures 6-8, are very similar in shape to the measurements reported by Page et al. (6). The best-fit lines of Weibull PDF for these samples have values of  $c$  greater than 1 and the values of  $c$  for these samples usually increase somewhat as the wet pressing pressure is increased. We believe that the value of  $c$  for a normal sheet is in the range of 1.4 to 1.8.

The interval size at which the measured free fibre length was grouped significantly influences the 'smoothness' of its frequency distribution. The greater the interval size, the smoother the distribution, but this will also reduce the points available for the comparison with the predictions made by Equation 1. In this study we arbitrarily chose a 5- $\mu\text{m}$  interval to group the measured free fibre length for fitting the data with Equation 1. A 20- $\mu\text{m}$  interval was used when the distributions were plotted in order to smooth the data. As shown in

Figures 6-8, Equation 1 fits distributions of the free fibre lengths reasonably well.

The measured probability in each interval (interval size is 20  $\mu\text{m}$ ) for all of the samples used in this study is plotted in Figure 10 against the corresponding probability predicted by Equation 1. As shown in Figure 10, the measured values and predicted values show very good correlation with a correlation coefficient of 0.97. This means that Equation 1 can describe the distribution of the free fibre length very well.

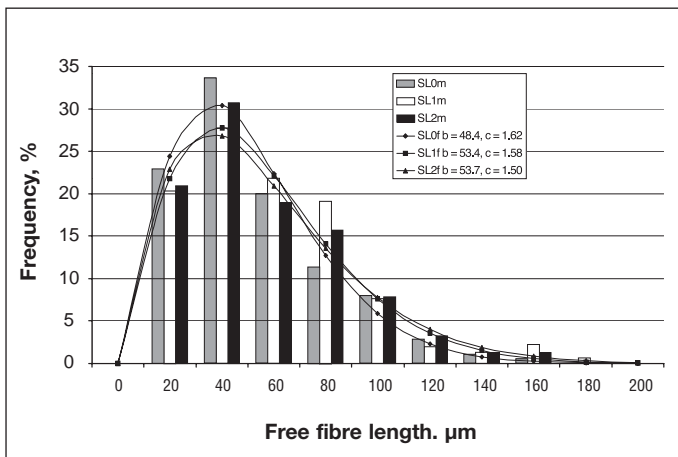
The out-of-plane deflection angles of all the fractions used in this study show no regular trend with the pressing intensity (Table 1). It can also be shown that the distributions of the out-of-plane deflection angle of samples made from the same pulp fraction but pressed at different pressing levels are very similar and show no regular trends with pressing intensity. These results are consistent with the results found in our previous study that were measured by a different method (15), but are inconsistent with Gorres and Luner's study (16), in which an assumption was made that the fibre deflection increases with increasing pressing pressure. If the out-of-plane deflection angle of the fibre segment stays constant, the deflection distance of the fibre segment will be reduced as the pressing intensity is increased. This is because the free fibre segment length has been reduced as the pressing intensity is increased. This movement will provide a densification effect for the paper structure in wet pressing.

Based on the experimental results of the distribution of the free fibre length, the number of fibre-fibre contacts per unit length of fibre, the percentage of full contacts and the distribution of the out-of-plane angle, a model structure of

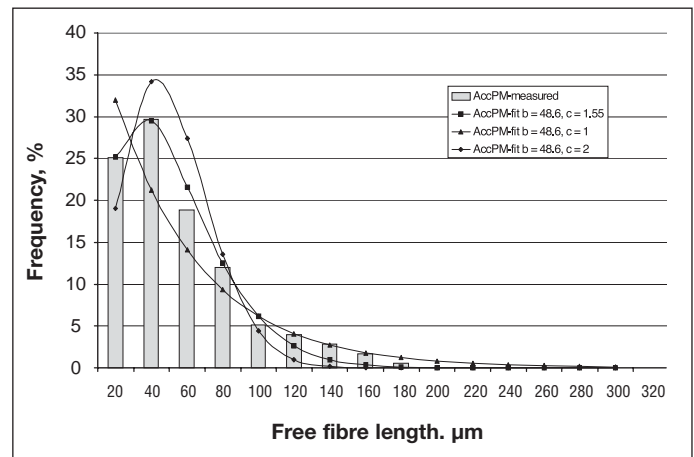
**Table 1**  
Summary of the average results of the geometrical parameters of paper structure.

Sample	$N_c^*$ (no./mm)	Full contact (%)	Partial contact (%)	Free Fibre length* ( $\mu\text{m}$ )	Out-of-plane angle* (degree)	Total length of fibre segment measured ( $\mu\text{m}$ )	Fibre width** ( $\mu\text{m}$ )	Fibre height** ( $\mu\text{m}$ )
AccP <sub>0</sub>	13.0±1.5	24	76	73.8±7.7	4.39±0.67	14978	31.6±1.3	13.7±0.7
AccP <sub>M</sub>	20.8±2.0	35	65	45.4±5.1	5.36±0.71	9594	34.3±1.4	11.9±0.6
AccP <sub>H</sub>	27.7±2.1	47	53	35.7±3.0	5.76±0.56	9504	36.6±1.5	9.7±0.4
RejP <sub>0</sub>	12.9±4.8	18	82	82.6±11.9	7.41±1.30	13340	29.5±1.2	15.8±0.8
RejP <sub>M</sub>	19.5±2.0	34	66	50.3±5.5	6.07±0.86	9281	32.7±1.3	14.0±0.7
RejP <sub>H</sub>	28.8±2.6	44	56	35.8±4.1	6.75±0.96	6862	34.9±1.3	11.2±0.4
SL <sub>0</sub>	23.4±2.4	48	52	42.1±4.2	4.30±0.63	8794	31.0±1.2	11.2±0.5
SL <sub>1</sub>	22.2±1.9	53	47	45.6±5.2	4.62±0.65	7528	31.8±1.2	12.3±0.6
SL <sub>2</sub>	22.5±2.1	50	50	45.4±5.0	6.35±2.27	8212	33.2±1.3	10.2±0.4

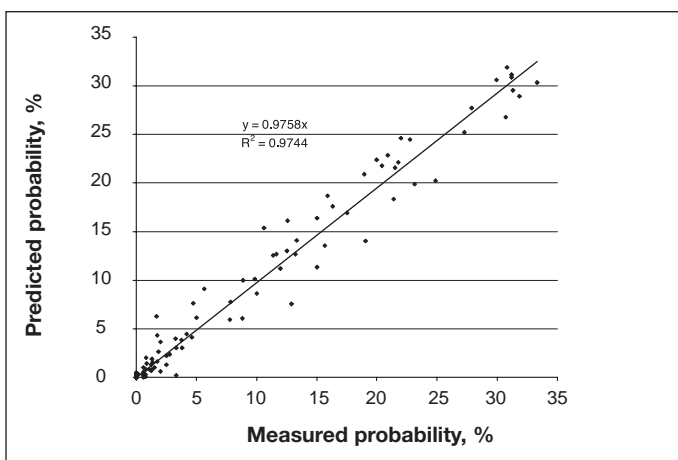
\* ± is 95% confidence interval. \*\* Fibre width and height were measured in sheets and fibre width has been corrected by the angle that the fibre sitting to the cross-sectional surface of the sample.



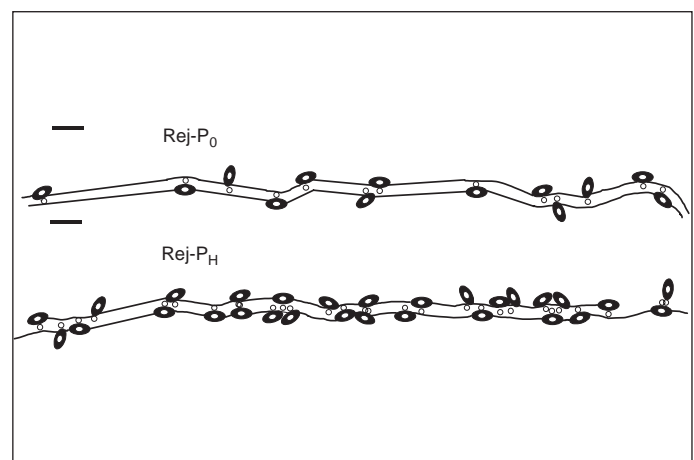
**Fig. 8** Frequency distribution of free fibre length of samples of fractions with different fibre length



**Fig. 9** Fit lines with constant  $b$  value but different  $c$  values for sample  $AccP_m$



**Fig. 10** Correlation of measured free fibre length distribution probabilities against the probabilities predicted from Equation 1.



**Fig. 11** Model structure of fibre-fibre contacts. \*The size bar is 50  $\mu\text{m}$ .

fibre-fibre contacts for a fibre segment of any of these samples can be reconstructed. Examples of such model structures for samples  $RejP_0$  and  $RejP_H$  are illustrated in Figure 11, in which the small circles along the fibre axes represent points between which the free fibre lengths were measured. The drawing here is to scale, showing the average situation of fibre-fibre contacts formed along a fibre segment of length 1 mm in those two samples. Clearly, in sample  $RejP_0$ , the arrangement of fibre-fibre contacts along the fibre segment is not uniform. There are some long free fibre segments. After it was pressed, the number of fibre-fibre contacts increased and the arrangement of the contacts became more uniform. The number of long free fibre segments was reduced dramatically. These model structures provide a direct view and better understanding of the structure of fibre-fibre contacts in paper, and these data can also provide important information for simulation studies of the load distributions along a fibre.

## CONCLUSIONS

The main conclusions from this work are:

- A new technique for measuring the properties of fibre-fibre contacts directly in paper has been proposed and has been used successfully. It is the first technique that can determine all of the parameters associated with the fibre-fibre contacts.
- It seems that fibre cross-sectional shape has no significant effect on the frequency of fibre bonding along a fibre and the distribution of the free fibre length. However, the accepts fibres (the thin walled fibres) tend to form a higher percentage of full contacts in a sheet.
- The distribution of the free fibre length is narrowed and shifted to a lower value range by wet pressing. The bonding frequency along a fibre is increased, while the free fibre length is reduced as the pressing intensity is increased. There is no significant difference in the number of

fibre-fibre contacts in the handsheets made from the accepts compared with those made from the rejects.

- The results obtained in this study suggest that fibre length alone has no effect on the properties of fibre-fibre contacts.
- The out-of-plane deflection angles of the free fibre segments have no regular trend with the pressing intensity for both the handsheets made from the accepts and from the rejects. However, wet pressing has reduced the out-of-plane deflection distance since the free fibre length has been reduced.
- The distribution of free fibre length for a normal sheet is not negative exponential. A two-parameter Weibull probability density function seems to adequately fit the data. .
- It has been demonstrated that model structures of fibre-fibre contacts can be reconstructed using the data measured by the new technique.

## ACKNOWLEDGEMENTS

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## CONCLUSIONS

Trials showed that the temperature responsive properties of a new group of hydrophobically modified starches could be used to achieve improved water resistance. The investigated starch grade was partly insoluble in water already at a very low degree of substitution with respect to hydrophobic groups. The hydrophobic modified starch gave a somewhat better flexographic printing result in terms of print density and white dots in full tone printing. This was especially noticeable when the starches were applied at high temperatures. For the oxidised starch it was evident that the best conditions may be surface treatment at room temperature, although the temperature effect was small and the results were not statistically significant.

Estimation of spreading of water on surfaces treated with starch can be very important for understanding the interaction with water-based printing inks on the same surface. Surfaces treated with starch gave lower mottling than untreated surfaces for all areas. Treatment with the hydrophobic starches gave better water protection with no increased tendency towards mottling.

The new hydrophobically modified starches have a potential to simultaneously improve print quality and water repellence.

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