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Tausif, M orcid.org/0000-0003-0179-9013, Duffy, B, Carr, H orcid.org/0000-0001-6739-0283 et al. (2 more authors) (2014) Three-Dimensional Fiber Segment Orientation Distribution Using X-Ray Microtomography. Microscopy and Microanalysis, 20 (4). pp. 1294-1303. ISSN 1431-9276

https://doi.org/10.1017/S1431927614000695

Complete Title: Three-dimensional Fibre Segment Orientation Distribution Using X-ray Microtomography

Brief Title: 3D fibre segment orientation distribution by XMT

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Abstract

The orientation of fibres in assemblies such as nonwovens has a major influence on the anisotropy of properties of the bulk structure and is strongly influenced by the processes used to manufacture the fabric. To build a detailed understanding of a fabric's geometry and architecture it is important that fibre orientation in three dimensions is evaluated since out of plane orientations may also contribute to the physical properties of the fabric. In this study, a technique for measuring fibre segment orientation as proposed by Eberhardt and Clarke (Eberhardt & Clarke, 2002) is implemented and experimentally studied based on analysis of x-ray computed microtomographic data. Fibre segment orientation distributions were extracted from volumetric X-ray microtomography datasets of hydroentangled nonwoven fabrics manufactured from parallel-laid, cross-laid and air-laid webs. Spherical coordinates represented the orientation of individual fibres. Physical testing of the samples by means of zero span tensile testing and z-directional tensile testing was employed to compare with the computed results.

Key words: orientation distribution, fibre, nonwovens, three-dimensional, X-ray microtomography, structure, hydroentanglement

1 Introduction

Nonwovens are fibrous assemblies where, similar to other products such as paper and composites, the spatial arrangement of fibres in the structure and their properties affect the physical properties of the assembly (Backer & Petterson, 1960; Narter, et al., 1999). The fibre orientation distribution (FOD) is of paramount importance as the placement of fibres cannot be individually controlled during manufacturing and the fabric cannot be represented by a geometrically repeating pattern. FOD is one of the key properties of a fibrous web or fabric and influences its mechanical behaviour (Xu & Ting, 1995), fluid transport (Rawal, 2010), pore size distribution (Rawal, et al., 2010), resin impregnation and the translation of fibre properties to composite product properties (Frank & George, 1998) and tissue growth on nonwoven scaffolds (Edwards, et al., 2010).

The structural characterisation of nonwovens in three dimensions is important as two fabrics having similar FODs in-plane can still behave quite differently if they possess dissimilar out-of-plane orientations. Substantial research of nonwoven fabric structure has been conducted based on analysis of two-dimensional (2D) images and measurement of orientation by means of binary image processing. One of the most basic 2D techniques to characterise FOD is by manual tracking of fibre segments. Other 2D techniques that have been developed include liquid-migration pattern analysis, zero-span tensile testing, laser diffraction analysis, light reflection and light refraction analysis, x-ray diffraction analysis, electrical measurements (Gong & Newton, 1996), direct tracking (Pourdeyhimi, et al., 1996b), flow-field analysis (Pourdeyhimi & Dent, 1997), Fourier transform (Pourdeyhimi, et al., 1997) and Hough transform (Pourdeyhimi & Kim, 2002). Progress has also been made in the evaluation of 3D FOD. These techniques include cross-sectioning of reinforced fibrous composite and analysis based on assumptions about the state of orientation (Blanc, et al., 2006), serial sectioning imaging (such as Digital Volumetric Imaging) (Shim, et al., 2010), confocal microscopy (such as confocal reflection/fluorescence microscopy) (Jawerth, et al.) and x-ray computed microtomography (XMT) (Eberhardt & Clarke, 2002; Yang & Lindquist, 2000). A detailed description of XMT is given by Stock (Stock, 1999). One of the advantages of XMT is that it enables non-destructive 3D study of the internal structure, minimising fibre disturbance and involves the collection of shadow images by rotating a sample at discrete angular increments before reconstructing each individual cross-section. An overview of reconstruction techniques for fibrous assemblies such as nonwovens is given by Eberhardt and Clarke (Eberhardt & Clarke, 2002).

Previously, 3D fibre orientation distributions obtained from analysis of 3D data has been explored in relation to the characterisation of collagen matrices (Wu, et al., 2003), fibre reinforced composites (Clarke, et al., 1995; Eberhardt & Clarke, 2002), bonded fibre networks such as those produced by solid state sintering (Tan, et al., 2006) as well as nonwovens (Eberhardt & Clarke, 2002; Yang & Lindquist, 2000) by employing various techniques.

Lux et. al. employed XMT to characterise local orientation of wood-based fibrous media by mathematical morphology and computed in- and out-of-plane fibre orientations.(Lux, et al., 2011) In another study, mathematical morphology tools were used to characterise local orientations, in few samples directions, from XMT data of carbon fibre reinforced composites. (Altendorf & Jeulin, 2011) Axelsson used segmentation to track the individual cellulosic fibres in XMT scan of cellulosic fibre reinforced composite. (Axelsson, 2007) Local orientation in XMT data of short-fibre-reinforced concrete and carbon- fibre-reinforced silicon carbide composites were computed by using structure tensor and X-ray local transform methods.(Krause, et al., 2010) Relative bonded area, as a measure of fibre-to-fibre bonds was studied in XMT images of fibrous materials by employing image processing methods (Malmberg, et al., 2011) and the accuracy was dependent on the initial lumen segmentation.(Malmberg, et al., 2011; Wernersson, et al., 2009) Fibre length and fibre orientation distribution was measured from XMT scan of short fibreglass reinforced phenolic foam and authors highlighted the need of an automatic computer program for the identification of 3D objects and measurements of geometric characteristics. (Shen, et al., 2004) A micro-compression rheometer was mounted during XMT data acquisition to collect the 3D images during compression loading on specimen for in-situ microstructure characterisation. Image analysis sub-routines were employed to characterise fibre orientation, Geometrical torsions and curvatures and number of fibre-to-fibre contacts. (Latil, et al., 2011) It is particularly useful to be able to characterise the 3D FOD of fibrous assemblies but comparison of the computed results with in-plane and out-of-plane mechanical properties can be useful for indirect yet simplistic comparison of the computed results. Thus, the current study aims to evaluate 3D FOD from non-invasive and non-destructive XMT imaging and to compare the results with experimental data for three different web formation methods consolidated by hydroentanglement. The results are represented as a simple extension from 2D to 3D FOD for intuitive understanding of microstructure of nonwoven fabrics.

2 3D Fibre segment orientation distribution

Komori and Makishima (Komori & Makishima, 1978) suggested that fibre orientation can be approximated by hypothetical subdivision of bent fibre lengths into short and straight segments along the fibre length, where the size of subdivision affects the evaluated direction and orientation distribution of these segments. The orientation vectors will be parallel to the straight fibre segments and coincide with the tangent to the fibre shape. In 2D, each pixel is connected to eight neighbours while in three dimensions each voxel is connected to twenty-six neighbours that make the conventional methods of skeletonising, based on line detection algorithms, computationally very intensive. Eberhardt and Clarke (Eberhardt & Clarke, 2002) used local orientation vectors to trace fibre paths for nonwoven and short-fibre reinforced composite samples. Their algorithm has been followed in the current work to determine the fibre segment orientation distribution. The algorithm was originally applied in four steps:

1. Vectors are identified representing the most probable orientation.
2. The vectors that are on the fibre centre lines are identified.
3. The identified vectors are connected.
4. Smooth curves are fitted to each fibre.

However, in the current study, only the first two steps were followed because the FOD can be estimated as an orientation distribution of vectors for fibre segments. The orientation of each fibre segment (a) of length (l) can be represented in spherical coordinates using two angles [θ(a), φ(a)], Figure 1, where θ is the orientation angle between the projection of a fibre segment on the xy-plane with respect to the x-axis while φ is the angle of a fibre segment with respect to the fibre segment projection on the xy plane. Figure 2 illustrates the process of acquiring local orientation vectors from a fibre sample in 2D. Figure 2a shows an example of a fibre F and two tangent vectors t_1 and t_2. Figure 2b shows the digital representation of the fibre; where the original fibre is overlaid on the pixel representation. Local orientation vectors, i.e. the tangent vectors t are computed discretely from this representation by applying a kernel of size l to the neighbourhood pixels of a pixel on the fibre. For one pixel on the fibre, the distribution of mean chord intensities for each angle θ is represented in Figure 2c. The scanned fibre lies approximately along the angles with the maximum mean chord intensity peaks as seen in Figure 2c. There are two ways of fitting a tangent vector to a fibre segment as shown in Figure 2a which correspond to two peaks in orientation distribution at a difference of π; this is illustrated in Figure 2c where θ is distributed over the range of 0 to 2π. However, in this paper these two directions are considered as equivalent. Hence, the range of [θ(a), φ(a)] angles can be presented by 0 to π and scanning along the machine direction, a semicircle may be used as the kernel so all fibre orientation/tangent vectors will be oriented in the machine direction. This process can be extended in the three dimensions to account for φ by using slices that are above and below the plane being computed; therefore a hemispherical kernel was used.

For the current work, normalised vectors were sufficient as interest lies in the orientation of the fibre segments only. This method is an intuitive extension of the existing 2D FOD where fibrous assemblies are treated as planar assemblies and θ presents the in-plane (xy) FOD. As most nonwoven manufacturing methods produce fabrics with a degree of preferential orientation of fibres (Neckář & Das, 2011) for ease of visualisation, the range of distribution of θ and φ can be presented as (-π/2, π/2) where $\theta = 0^0$ for fibres aligned to MD and $\varphi = 0^0$ when a fibre segment is parallel to xy plane (in-plane).

3 Materials and Methods

3.1 Sample preparation

Parallel-laid and cross-laid webs were manufactured using 0.5 m wide laboratory worker-stripper carding machine while air-laid webs were prepared using a lab-scale unit comprising nipped feed rollers, a pinned cylinder and a rotary condenser on to which fibres were deposited to form the web. Fibre and sample specifications are given in Table 1. All webs were hydroentangled using identical process conditions, once on each side using two injectors fitted with a single row of 110 µm diameter jet strips at a conveyor speed of 5 m min^{-1}. Further process details are given in Table 1. After hydroentangling samples were through-air dried.

3.2 X-ray microtomography

Samples were laser cut into swatches of 12 mm x 5 mm size to minimise potential structural disturbance due to mechanical cutting. Micro-focused X-ray microtomography (Phoenix X-ray nanotom) was utilised to collect three-dimensional imaging data for each sample. Scanning was performed at a resolution of 2 μm per voxel. The voltage, current and irradiation time were optimised to obtain good contrast and were 80 kV, 100 μA and 1000 ms, respectively. The angle increment for scanning samples was 0.25° per step for 360°, providing 1440 cross-sections for reconstruction. The projection images obtained were computationally reconstructed to produce a 3D spatial image using VG Studio Max. The dimensions and volume of scanned samples are given in Table 2. An exemplar of reconstructed XMT images along with sections in principle directions is shown in Figure 3.

3.3 Algorithm implementation

The software application for segmentation and image processing of XMT data was written in the C++ programming language. In addition, a graphical user interface (GUI, Figure 4a) was developed in the Qt and OpenGL graphics Libraries. All datasets were processed on a Dell Precision T1500 64-bit platform, Intel Core i7-870 CPU with 4 cores (8 logical processors/threads) at 2.93 GHz clock speed and 6 GB RAM.

The FOD histograms were presented in 15^0 bins for both θ and φ, averaged for negative and positive groups of similar angles to obtain symmetry around 0^0 by employing Matlab and SPSS. It is already reported in the literature that mechanical properties are similar at equal angular difference from 0^0 (Gong & Newton, 1996; Hearle & Ozsanlav, 1979). Sampling artefacts and noise are always present in XMT data due to photon statistics (Davis & Elliott, 2006) . To accelerate data processing, thresholds were applied to the voxels using the frequency distribution of voxel intensities (Figure 4b) to avoid computation on noisy and background voxels and the segmented voxels were termed “active voxels”. The voxels between these thresholds then correspond to the locations of the fibre segments as in Figure 4c. The selection of an appropriate segmentation range is discussed further in section 3.4.

Once the segmentation was performed, the dataset was processed slice-by-slice by streaming from the hard disk drive (HDD). A block of slices of size L corresponding to the neighbourhood of the mean chord intensity computation was streamed for each slice and processed in parallel. In addition to this, when computing the orientation vectors at each voxel, a hemispherical kernel was used, which was aligned with the machine and cross directions in the XMT scans essentially cutting the number of computations per voxel in half. A block L was divided among the available threads using a simple load-balancing scheme based on the number of active voxels in each slice. This ensured that the work for each thread was evenly distributed and no thread would remain idle for extend periods of time. The parallel software was implemented using QThreads from the Qt library.

3.4 Variables for computation

The implementation allows the user control over three input variables in the software: orientation vector length, segmentation range and angle interval/step size. These variables are likely to affect the computation times and influence the accuracy of results. A preliminary study was carried on the parallel-laid sample (Table 1) to study the effect of aforementioned three variables.

The selection of an appropriate length of orientation vector is important to avoid misleading results. The ideal approach would be to calculate mean fibre segment length between bonds (Gong & Newton, 1996) but it is an enormous task itself. A more practical approach has been reported in the literature where the angle distributions for a range of lengths are computed and stabilisation of distribution aids the selection of appropriate orientation vector length (Gong & Newton, 1996; Pourdeyhimi, et al., 1996a). Theta (θ) and Phi (φ) were computed at a range of vector lengths as shown in Figure 5a and Figure 5b, respectively. The results at 5 voxels (10 µm) were misleading due to the fact that mean fibre diameter for PET is 13±2.3 µm and interest lies in the orientation vector, which is tangent to the straight fibre segment. The distributions of angles were found to stabilise at ≥ 50 voxels, hence further computations were performed at 50 voxel long vectors (i.e. 100 µm fibre segments).

The parallel-laid samples were segmented at three different segmentation ranges (0.60%, 1.62% and 2.84% of active voxels), Figure 6, and results are depicted in Figure 7a and Figure 7b for Theta (θ) and Phi (φ), respectively. Note that the low values of active voxels percentages for each "range" confirm that many noisy pixels were present in the original dataset. It is evident that segmentation range does not affect the computation of angles as even low segmentation is representative of the original dataset. Note that the percentage active voxels varies from dataset to dataset and the selection is subjective to the user's knowledge of the scanned fabric. In case more than one fiber are employed in a study and employed fibers exhibit different densities during the XMT, such fibers could be studied individually by thresholding fibers on the basis of respective voxel densities.

The angle interval/step size directly affects the computation time; the number of steps was calculated by a 360°/angle interval. As data is eventually presented in 15° bins, hence the parallel-laid sample was processed at 1°, 5° and 15° intervals to see the effect of angle interval on Theta (θ) and Phi (φ) in Figure 8a and Figure 8b, respectively. Apparently, there was no major difference but for Phi (φ) even small difference can be expected to be important as most of the fibres are laid in-plane and a small difference of through thickness migration of fibre segments can be influential. Hence, for good practice, all subsequent computations were carried out at 5° intervals. The computation times for all three variables are given in Table 3.

3.5 Comparative tensile testing

Tensile testing of was carried out to test the correlation of in- and out-of-plane mechanical properties with Theta (θ) and Phi (φ), respectively, for comparison with results computed from the XMT datasets.

3.5.1 Zero span tensile testing

Zero span tensile testing (ZST) tests results can be linked to in-plane fibre segment orientation distribution (Pourdeyhimi, et al., 1999). Since the gauge length is zero during ZST, fibre segments mainly aligned parallel to the test direction, gripped between the two jaws, are the load bearing component and thus the major contributor to breaking strength (Batchelor, 2003; Kallmes, 1969). The contribution of individual fibres to the tensile strength of the fibre assembly, subject to all other factors being constant, is proportional to the $\cos^2\gamma$, where γ is the angle between the fibre segment and the direction of tensile force. It can be seen that as the angle γ increases, the fibres are less aligned with the direction of force and the contribution of inclined fibres significantly decreases. Hence, ZST was performed using a universal strength testing machine to correlate Theta (θ), computed from the XMT data, for comparison with in-plane tensile properties of the fabrics. Specimen strips of 50 mm long and 25 mm wide were cut and tested at an elongation rate of 100 mm min^{-1} at zero gauge length. As is already known (Gong & Newton, 1996; Hearle & Ozsanlav, 1979) and was confirmed in this study, the distribution shows a symmetry around 0°, therefore ZST was determined for three specimens of each sample for 0°-90° at 15° intervals to compare with the value of θ computed by the fibre segment detection algorithm.

3.5.2 z-directional tensile strength

The out of plane orientation distribution (phi, φ) of fibre segments directly affects the z-directional strength of a fabric. A modified version of the BS ISO 15754 standard method (Tausif & Russell, 2012) was utilised to characterise z-directional strength of the samples.

Prior to testing, samples were subjected to a compression of -280 KPa at 5 mm min^{-1} followed by extension at a rate of 400 mm min^{-1}.

4 Results and Discussion

4.1 Results for Theta (θ)

Plots for θ and ZST are given in Figure 9 for the parallel-laid, cross-laid and air-laid samples; the variation exists, within acceptable limits, in the sample and the characteristics shapes of the respective distributions persist. The computed distributions of θ based on the analysis of XMT data showed a high correlation with the ZST data for parallel- and cross-laid samples; the correlation coefficients being 0.953 and 0.910, respectively. The comparison between these two distributions is a recognised method of correlating FOD and mechanical properties (Kallmes, 1969). Virtually no correlation was found between the in-plane fibre orientation and tensile strength for the air-laid samples where the correlation coefficient was 0.137. In ideal air-laid webs, the probability of orientations at all angles would be the same and a similar trend was observed in Figure 9c. Parallel-laid and air-laid samples exhibited characteristically shaped distributions as extensively reported in the literature (Hansen, 1993). Because the cross-laid sample was produced with a small lapping angle of only 2° the distribution appeared close to the characteristic distribution of a transverse-laid sample.

4.2 Results for Phi (φ)

Figure 10 plots the values of Phi for each of the parallel-laid, cross-laid and air-laid samples and the intra-sample variation is within acceptable limits. After hydroentangling, the majority of fibre segments in air-laid, cross-laid and parallel-laid fabrics remained oriented in-plane, which is consistent with results reported by Lux et. al. However, differences were apparent in the frequency of out-of-plane fibre orientations according to the method of web formation. To more clearly elucidate these differences, the fibre segment orientation frequencies obtained for each sample were compared for values of $\varphi = \pm$ 61-90°, Figure 11. The extent of out-of-plane fibre segment orientation was confirmed as air-laid > cross-laid > parallel-laid. It is known that air-laid webs can exhibit greater through-thickness fibre orientation as a result of the mode of fibre deposition during web formation, but also, differences in the out of plane fibre segment orientation between the three fabrics could reflect the way in which fibres within each web behave during hydroentangling. Consider the orientation of fibres in the web relative to the impinging water jet curtain. In the air-laid webs, a greater number of fibre ends were present compared to the parallel-laid and cross-laid webs because of the shorter fibre length and these fibre ends are readily deflected out-of-plane by the impact forces and internal vortices. In a parallel-laid web, fibres were preferentially aligned in the machine direction perpendicular to the water jet curtain and the extent to which a jet is likely to impinge upon a fibre along its full length will be influenced by their relative position. In a cross-laid web where fibres were predominantly aligned almost parallel to the water jet curtain, a more extensive jet impingement of a fibre along its length would be anticipated. Note that in the present experiment only one injector was utilised on each side of the web so that any variations in fibre segment deflections as a result of the spatial position of the jets relative to the incoming fibres are likely to be exaggerated.

The parallel- and cross-laid webs, which were of identical fibre composition and were produced using similar conditions, exhibited a large difference in z-directional tensile strength as well as the frequency of out-of-plane fibre orientations, $\varphi = \pm$ 61-90°. The ZTS of the hydroentangled cross-laid fabric was higher than the parallel-laid and this may be a reflection of the higher out-of-plane fibre segment orientation in the former, Figure 10.

4.3 Limitations of the data capture method

A limitation of the data capture method used is the maximum spatial resolution during tomography. For the algorithm to produce accurate fibre orientation measurements a number of fibre characteristics (fibre diameter, fibre cross-section, fibre packing fraction, fibre curl and fibre crimp) must be considered that may impact accuracy in reconstructing fibre orientation information from the volume dataset.

If fibre diameter decreases to less than the maximum spatial resolution, more than one fibre contributes to a single voxel giving an aggregated measurement of fibres. Furthermore, as discussed in section 3.4, the length of the orientation vector should be greater than the diameter of the fibre to avoid any misleading results. Fibres with approximately circular cross-sections were used in this study, but other fibres can have a multi-lobal or crenulated cross sectional morphology. Since, only the orientation of fibre segments is calculated per voxel, the cross-sectional shape of the fibre will not affect the accuracy of results. A high packing fraction of fibres increases their inter-connectivity and if the sampling resolution is too low, the XMT scanner will aggregate these fibres together in the scan. This will impact the algorithms accuracy such that it may be unable to distinguish closely packed fibres. Fibre curl and crimp can further complicate the analysis. The orientation distribution of fibres in this study are of smaller segments, hence fibre curl is not likely to occur in the samples under study. In cases where fibre crimp is present, the length of the orientation vector should be greater than the crimp length to avoid misinterpretation of fibre segment orientation distribution.

To summarise, issues with algorithm accuracy are introduced when the sampling resolution of the scanner is too low to accurately capture features in the fibre assembly. Put simply, information is lost, and this phenomenon is well known in Information Theory as the Nyquist sampling limit (Oppenheim, et al., 1999). However, in this study samples were scanned at sufficiently high spatial resolution of 2 μm per voxel with respect to the mean fibre diameter of 12 μm for lyocell and 13 μm for the PET fibre.

4.4 Limitations of the algorithm

In this paper we implemented the fibre segment orientation estimation scheme presented by Eberhardt and Clarke(Eberhardt & Clarke, 2002). This approach estimates fibre segment orientation using a brute force search of the spherical neighbourhood around each voxel. The algorithm performs a linear search in θ and φ of the spatial domain for the most probable orientation vector and computes a discrete approximation of the local orientation vector. Additionally, the algorithm is bounded by the selection of the step size in θ and φ as well as the length of l. The number of additions, multiplications and trigonometric function calls per voxel vary and trigonometric function calls can be eliminated by pre-computing angles. Therefore, every vector must be checked and compared to the current maximum intensity vector peak and this strategy offers no method of early termination. In addition to the slow speed of searching the spatial domain, the brute force method requires additional memory to compute the orientation vectors even with a slice-by-slice strategy. Additional slices must be streamed from disc in the neighbourhood of L to compute the orientation vectors. This requires more disc accesses, which is slow and the number of disc accesses varies with number of processors. Additional memory is required for computing the most probable orientation when there are multiple same maximum peak intensity values. The existing algorithm for fibre segment detection followed in this particular study is not scalable for larger data sets.

5 Conclusions

An algorithm developed by Eberhardt and Clarke (Eberhardt & Clarke, 2002) has been extended to enable computation of the 3D fibre segment orientation distribution (FOD) in spherical coordinates (θ , φ); where θ represents the in-plane and φ represents the out-of-plane fibre segment orientations. The selection of a suitable orientation vector length is important to avoid misleading results and computation times can be controlled by appropriate selection of variables (vector length, segmentation range and angle interval). Three primary fibrous webs (air-laid, cross-laid and parallel-laid) were subsequently hydroentangled to compute the 3D FOD and results were compared with the results of tensile testing, which gave good agreement. Zero span tensile testing and z-directional tensile testing were found suitable physical tests for correlating computed θ and φ, respectively. The majority of the fibre segments remained oriented in-plane and differences in out-of-plane deflections for $\varphi = \pm$ 61-90° could be explained by differences in the microstructure of fabrics. Air-laid webs exhibited better entanglement due to inherently random fibre deposition and more fibre ends available for out-of-plane deflections. Cross-laid webs resulted in higher z-directional strength owing to more frequent out-of-plane fibre segment orientations compared to parallel-laid webs, potentially due to the alignment of fibres in the web relative to the impinging water jet curtain during hydroentanglement. The computation of 3D FOD has been successfully demonstrated and this technique can be applied to a variety of fibrous assemblies to study the effect of process variables on fabric geometry. The followed algorithm of XMT data processing can be further improved to allow large datasets to be analysed.

6 Acknowledgements

The authors gratefully acknowledge the support of the University of Engineering and Technology, Lahore, Pakistan (Faisalabad Campus) in providing a scholarship to one of the authors (MT) and of Institute of Particle Science and Engineering, University of Leeds for their help in XMT imaging.

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Figure Captions

Figure 1: Representation of a fibre segment (vector) in Cartesian coordinates by spherical coordinates. θ and φ are presented as (-π/2, π/2) where $\theta = 0^0$ for fibres aligned to MD and $\varphi = 0^0$ when a fibre segment is parallel to xy plane (in-plane).

Figure 2: Example of the description of the intensity profile for a fibre segment at θ (0-2π**)** **(a)** Fibre F with two tangent vectors t_1 and t_2 **(b)** Digital representation of the fibre **(c)** The distribution of mean chord intensities for 0-2π (Consult online copy for coloured version)

Figure 3: An exemplar of reconstructed XMT image for hydroentangled parallel-laid sample along with sections of three principle directions

Figure 4: **(a)** GUI interface of the software with a XMT slice loaded for the hydroentangled parallel-laid sample (Table 1, Figure 3) **(b)** histogram peaks for a slice loaded in Figure 4a **(c)** Active voxels segmented on the basis of histogram peaks in Figure 4b

Figure 5: **(a)** Theta (θ) and **(b)** Phi (φ) computed at a range of voxel lengths (05→80) for the hydroentangled parallel-laid sample at 5° interval and 1.62% active voxels. (For clarity, the reader is referred to the web version of this article for the figure in colour.)

Figure 6: A characteristic CD-Thickness (length-width) slice of the hydroentangled parallel-laid sample with different ranges of segmentation (% active voxels) **(a)** 0%, **(b)** 0.60%, **(c)** 1.62%, **(d)** 2.84%.

Figure 7: (a) Theta (θ) and **(b)** Phi (φ) computed at three different segmentation ranges (% active voxels) of the hydroentangled parallel-laid sample for an orientation vector length of 50 voxels and angle interval of 15°.

Figure 8: (a) Theta (θ) and **(b)** Phi (φ) computed at 1°, 5° and 15°of hydroentangled parallel-laid sample for an orientation vector length of 50 voxels and 0.60% active voxels.

Figure 9: Zero span tensile strength of physical samples and Frequency % histogram of Theta (θ) is computed from the 3D XMT datasets for **(a)** parallel- **(b)** cross- and **(c)** air-laid hydroentangled samples

Figure 10: Plots for Frequency % computed from the 3D XMT data plotted with φ for the hydroentangled samples: (a) parallel-laid (b) cross-laid (c) air-laid

Figure 11: Out-of-plane fibre segment deflections (φ = ±61-90°) and z-directional tensile strength (n=5 specimens) for air-, cross- and parallel-laid hydroentangled samples. Note that air-laid sample has different fibre specifications so that its tensile strength cannot be directly compared to the other samples (see Table 1)