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CHARACTERISATION OF THE 3D PAPER STRUCTURE WITH X-RAY SYNCHROTRON RADIATION MICROTOMOGRAPHY

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ABSTRACT

Paper is a complex three-dimensional network of fibres, pores and often fillers. The main goal of this study is to characterise its structure in a non invasive and non destructive way. In order to overcome the limitations of 2D measurements, Synchrotron Radiation microtomography is used to visualise the samples. Coupled with appropriate processing tools, it allows a quantification of some structural characteristics on the samples. This is the main aim of this paper. Different articles relate the feasibility of such a study for paper samples: imaging at the European Synchrotron Radiation Facility (ESRF, France) in phase contrast [1] or in absorption mode [2] gives the opportunity to reach a pixel size smaller than a micron which can not be obtained with classical tomographs [3]. A first step is to visualise the structure then, structural parameters are extracted from these 3D data [4]. However, this requires a segmentation technique adapted for application to typical paper samples that are constituted of three phases. The first step consists in the segmentation of the different phases, namely, air, fibres and fillers. The amount of each component may be evaluated. This was validated for both the porosity and the filler content. Furthermore, structural parameters were calculated from the binarised volumes. The comparison with the published results validates the calculation.

1 INTRODUCTION

The main goal of microtomography is to build a representation of the inner structure of a sample from radiographs in a non-destructive way [5]. This unique 3D imaging technique may be used for investigations on a large variety of materials: metal alloys, porous structure such as felt [6], biological samples such as bone. It overcomes limitations of traditional 2D imaging tools and allows access to three dimensional parameters when coupled with appropriated image processing tools. This tremendous technique is the perfect tool to characterise paper and board structures [1, 2, 7].

The paper is organised as follows: in section 2, Synchrotron Radiation microtomography is presented and a segmentation method is proposed; section 3 concerns the validation of the tools presented in the previous section; section 4 deals with examples of sample characterisation.

2 DATA ACQUISITION AND PROCESSING

The experimental set up and the sample preparation is first briefly described.

2.1 X-Ray Synchrotron Radiation microtomography in absorption mode

Compared to classical X-ray sources, the synchrotron facility provides an X-ray beam characterised by a high flux of photons (high brilliancy). This beam is tuneable in terms of energy and size. All the data presented in this work were acquired at ESRF on the ID19 beamline. On this beamline, the X-Rays are provided by a source which size is $60 \ \mu m$ (horizontal) by $10 \ \mu m$ (vertical). This small size of the source (<100 $\ \mu m$) combined with other outstanding features such as the small horizontal and vertical divergency of the source (<0.5 nrad) and large distance between the source and the sample (145 meters) leads to a very small angular extension of the source as seen from a point within the specimen, hence to a sizable lateral coherence of the X-ray beam.

All the presented results in this paper were performed with absorption

contrast technique. This method relies on the Lambert Beer law (Equation 1)

$$\int_{path} \mu(x, y, z)dz = -\ln\left(\frac{I_n(x, y)}{I_o(x, y)}\right)$$
(1)

The provided beam is monochromatic and parallel. The relationship between the intensity $I_n(x,y)$ transmitted after a path of length z in the sample, the incident intensity $I_o(x,y)$ and the projection of the linear attenuation coefficient μ , is straightforward and one-to-one. Assuming that this projection is known for a large number of angular orientations of the sample, tomographic reconstruction makes it possible to quantitatively map the distribution of $\mu(x,y,z)$. The linear attenuation coefficient μ depends, for given X-ray energy, only on the composition and the density of the material [4].

2.1.1 Experimental set-up

Figure 1 represents the scheme of the experimental set up that was used during the data acquisition part. A multilayer coatings for x-ray optical devices whose bandwidth (Δ E/E where E states for energy) is 10⁻² provides a monochromatic beam. Experimental experience suggests that an energy of 20.5 keV is the best choice to perform our acquisition. The sample is placed on a high precision rotation-translation stage that aligns the three main components: beam, sample and detector accurately (i.e. within 0.1 µm). Sample preparation is discussed in the following paragraph. A scintillator converts the transmitted beam into visible light which is recorded by a FReLoN camera (Fast Read Out Low Noise CCD detector) developed at ESRF [8]. According to the typical dimensions of fibres, a pixel size of 0.7 µm is chosen leading to a field of view of 1.4 mm × 1.4 mm. To avoid local-tomography



Figure 1 Scheme of the experimental set-up of ID19 beamline.

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drawback (reconstruction more noisy) the typical size of the samples is close to $1.4 \text{ mm} \times 1.4 \text{ mm} \times \text{paper_thickness mm}$.

To reach a satisfying reconstruction quality, radiographs are performed for 1500 angular positions evenly distributed over 180 degrees.

2.1.2 Sample preparation

A key component of the tomographic experiment is the preparation of samples. Several methods were tested. None of them kept the sample from moving or swelling. The method presented hereafter gives the best result. This means a strain of the sample of less than 1 μ m during the whole tomographic scan as determined from the comparison of the images for 0° and 180° of the scan.

The sample holder is a capillary. It is placed on the rotation-translation stage of the microtomograph. The sample is put on the top of this capillary. To avoid glue penetration into the sample of paper, a "Post-it®" was placed between the paper and the capillary. The "Post-it®" is glued to the capillary.

Setting the sample parallel to the top of the capillary is not always possible (Figure 2). This is in general due to the non-uniform repartition of the glue on the capillary. In this case, the vertical direction of the stack does not match to the thickness of the paper. Hence, 3D measurements will be biased.

Moreover taking into account that the paper is sensitive to its environmental conditions (temperature and humidity), the prepared samples are placed in the experimental room about twelve hours before imaging them.



Figure 2 Radiography (0.5 mm × 1.4 mm) and reconstructed slice (1.4 mm × 1.4 mm) for a printing paper. Visualisation of the tilt encountered during sample preparation.

2.1.3 Reconstruction

In practice, slices are reconstructed one after the other using a filtered-back projection algorithm. It is based on the Fourier slice theorem that relates the Fourier transform of a projection to the Fourier transform of the object along a single radial. Namely, the Fourier transforms of the recorded radiographs are gathered to yield an estimate of the 2D Fourier transform. Inverting this gives an estimation of the object [9]. Figure 3 represents an example of visualisation of data during the different steps: recording radiographs for different angular positions, validation of the radiograph set with the reconstruction of one slice, and reconstruction of the volume.



Figure 3 Different steps of the data acquisition of a blotting paper: radiograph at 0 degree (0.840 mm \times 1.4 mm), one reconstructed slice (1.4 mm \times 1.4 mm), and 3D visualisation of the reconstructed volume.

2.2 Data processing

2.2.1 Filtering

Despite the high quality of data obtained at ESRF in terms of signal to noise ratio [10], a numerical filter must be applied. A non linear filter, named anisotropic diffusion, is chosen since it allows inner regions to be smoothed whilst preserving edges [11]. A more elaborate version in 3D was implemented: the filter kernel is composed of a gradient term, which enables edge preservation, and a measure of 3D homogeneity, which increases the smoothing effect. More details can be found in [12]. Figure 4 illustrates the result of filtering.

The visual comparison of the two slices shows that the edges and shape of fibres are preserved; no benefit can be observed visually. The histogram's features are used to verify the efficiency of the filtering. In most cases, histograms of paper samples imaged with X-Ray Synchrotron Radiation micro-tomography present one mode which is typical of a single phase material. This is due to the weak difference in absorption of X-ray between air and



Figure 4 Result of the denoising step. Slices ($1050 \ \mu m \times 490 \ \mu m$) are extracted from a handsheet. The histograms correspond to the slice above and represent the number of voxels for the different linear attenuation values.

cellulose. Applying the non linear anisotropic filter two or three modes appear and these are typical of the paper components: fibres, pores and fillers if present.

2.2.2 Segmentation: a seeded region growing method

Segmentation is the key point to extract quantitative measurements from data. Few segmentation methods have been applied to paper tomograms. One method based on global threshold [7], [13], [14] has been applied to filtered data. It is an easy method that gives correct results to separate pores from the other phase. However it is still sensitive to noise (Figure 6) and does not allow separation of fillers from fibres in the solid phase. Another method is proposed by Antoine [15]. This method combines several image processing techniques: filter, threshold, region growing, dilatation, etc. This method does not take into account the 3D information provided by the tomographic data since data are processed slice by slice.

In this context a 3D segmentation method was implemented [16]. The seeded region growing method is a segmentation method based on voxel aggregation. The voxels are regrouped according to a double criterion: a homogeneity criterion and an adjacency one. It usually takes into account the nature of the analysed data and represents the constraints the region must

satisfy. This process creates the region one after another. Furthermore each starting region called seed is submitted to an evolution phase. The final result is a partition of the data into regions R_1, R_2, \ldots, R_i . The quality of the obtained segmentation mainly depends on the choice of the seeds [17].

It has been arbitrarily decided that the region growing algorithm is applied to the fibres. Consequently the starting regions must be located inside the fibres. They are defined using a threshold upon which the quality of the segmentation relies. As mentioned before, the histogram of the denoised volume presents at least two peaks: the first one centred on 0 is related to the pores and the second centred roughly on a value greater than 1.1, in the presented example, is related to the solid phase (fibres and fillers). The voxels whose value is above the second peak belong to the fibrous part. The candidate voxel to a region is chosen in a $3 \times 3 \times 3$ neighbourhood. Then the growing criterion is applied to this region. This was chosen according to the nature of the data: some phase contrast may be noticed along most of fibres. A white fringe may be seen inside the objects and a black one inside the pores. This variation in intensity is used to define the homogeneity criteria (Equation (2)), explained as follow.

Let P be a voxel adjacent to the considered region R_i , M_a the local mean of the voxels of R_i 's border that are next to P and M_b the local mean of the voxels both of P and of R_i 's border that are next to P. If

$$\frac{|M_a - M_b|}{M_b} \le criterion_value \tag{2}$$

then P is included into region $R_{\rm i}$. This criterion based on a relative error of the mean intensity along the border of the objects preserves homogeneity within regions from an intensity point of view. The results of this segmentation applied to paper's data are illustrated in Figure 5. The segmentation achieves its goal from a visual point of view. This is confirmed by the superposition of borders of fibres obtained with the 3D region growing algorithm superposed onto the denoised slice.

More over this method is less sensitive to noise as shown in Figure 6.

This is a two parameter method: the local threshold and the criteria_value both depend on the type of objects to be segmented. The segmentation process can be applied to different kinds of papers. Different values of the parameters of the developed software may be used to separate fibres, fillers and pores. Figure 7 illustrates the separation of the three components of a printing paper filled with precipitated calcium carbonate and a Kraft paper filled with calcium carbonate.

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Figure 5 Result of the segmentation process for a paper board sample $(1.4 \text{ mm} \times 1.4 \text{ mm})$. The first slice (a) represents the denoised data, the second (b) the corresponding binarised one. On the last one (c), the borders of fibres obtained with the 3D region growing algorithm are superposed to the denoised slice.

We can see in Figure 7 the pores in black, the fibres in white and the fillers in grey. Moreover, we have a direct access to the localisation of the fillers as Figure 8 illustrates it.

A developed segmentation method allows the separation of the three main constituents of paper. The next step is to validate this method by evaluating different structural parameters on the binarised data.



Figure 6 Results of the segmentation process for a handsheet. The slice of the top is the slice segmented with the proposed method (a), and the bottom (b) slice is obtained with a global threshold. Slices are 1050 μ m × 490 μ m and the segmentation step is applied on denoised data presented in Figure 4. The blurred on the down left part of slices is due to the strain of the sample during acquisition.

3 VALIDATION OF THE DEVELOPED TOOLS: QUANTITATIVE RESULTS

Some elements are presented in this section to validate the computing techniques described above.



 $(504 \times 504 \text{ } \text{\mu}\text{m}^2)$



 $(504 \times 504 \ \mu m^2)$

original slice $(560 \times 560 \ \mu m^2)$

binarised slice $(560 \times 560 \ \mu m^2)$





a) Fibres and fillers spatial repartition

b) Filler spatial repartition

Figure 8 3D visualisation of the fibres and fillers repartition for a Kraft paper. The dimensions of the represented volume are 1.4 mm \times 1.4 mm \times 210 μ m.

Representative Elementary Volume 3.1

The first step is to check that the volume which is investigated is representative from the structure. The Representative Elementary Volume (REV) has therefore to be evaluated. The porosity is computed for different sized volumes [18] in order to evaluate the size of the REV. A different method based on statisitics [19] could also be applied. The graph in Figure 9a represents the evolution of porosity according to the size of a volume. For small volumes, the porosity is varying, and then it stabilises. The limit between these two behaviours allows computing the characteristic length, which is the cubic root



b)

Figure 9 Evaluation of the Representative Elementary Volume and porosity profile for a printing paper sample (COPY1).

of the limit volume. For this paperboard sample, it was evaluated to 140 μ m. Consequently the measured volume (1.4 \times 1.4 \times paper_thickness mm³) is representative of the volume in this particular case.

The graph in Figure 9b shows the porosity profile for the studied sample. The porosity profile shows the homogeneity of this printing paper. On the top, the porosity is influenced by the adhesive on the "Post-it®". On the

bottom slices the porosity variation is due to the contribution of ring artefacts. These artefacts are put in the same category as fibres when they are not located in the samples. It should be noticed that the porosity profile can be biased as the vertical direction during acquisition does not always correspond exactly to the thickness direction of the paper, as discussed above.

3.2 Repeatability of the methods and porosity evaluation

First of all, the repeatability of the developed methods, acquisition and segmentation, has to be tested. Therefore four samples of a commercial printing paper were randomly chosen from a paper ream and prepared for experiment. They have been imaged with X-Ray microtomography and processed with the same tools and same parameters during the denoising and segmentation steps. From a visual point of view, the segmentation achieves its goal as illustrated in Figure 10.

In Figure 10, we represent in white the solid phase (fibres and fillers) and in black the pores.

Different techniques have been proposed to define the surface of paper samples [20, 21]. They can be applied in our case on the top but not on the bottom of the samples as bubbles of the "Post-it®" are present. As the porosity profile of those samples (Figure 9) is characteristic of a homogeneous sample, the porosity can be evaluated for subvolumes extracted from the bulk. Each analysed volume is $1050 \ \mu m \times 490 \ \mu m \times 34.3 \ \mu m$ which is greater than the REV obtained for this kind of paper (140 $\ \mu m$). Table 1 shows the value of the porosity obtained for each sample.



Figure 10 Slices of samples segmented to study repeatability $(358.4 \mu m \times 358.4 \mu m)$.

Sample name	Porosity
COPY1	0.52
COPY2	0.57
COPY3	0.56
COPY4	0.49

 Table 1
 Porosity found using experimental data and signal processing tools

The mean is 0.53 ± 0.03 . It should be noticed that the size of the imaged volume corresponds to the size of a floc, which may explain the variation of porosity.

On the other hand, the porosity has been calculated with the parameters provided by the manufacturer using the following Equation (3):

$$\varepsilon = 1 - \frac{b_w}{t.\rho_c} \tag{3}$$

where ε , b_w , t and ρ_c , represent the porosity, the sheet basis weight (80 g.m⁻²), the thickness (111 μ m) and the cellulose density (1540 kg.m⁻³), respectively.

The porosity obtained with these data is 0.52 ± 0.02 . Both methods of porosity estimation give the same order of magnitude. Therefore, the processing methods are validated and may be used to estimate structural parameters.

3.3 Filler contents

This section deals with some measurements of filler contents.

We extract two samples from handsheets with known filler content. The first handsheets are composed of hardwood fibres and filled with calcium carbonate. The filler contents were 5%, 10% and 20% which represents the added amount. A reference sample, composed only of hardwood fibres, is also analysed. The seven samples imaged are processed with the same tools in order to denoise the volumes and to separate pores, fibres and fillers. The same parameters were used in each case. As seen in previous section, the borders of the segmented phase fit with the borders of the different components of the raw data. Therefore, the evaluation of the volume of each phase from the digital data, is carried out considering the number of voxels belonging to each constituents type. The definition of filler content (4) can be converted for digital data into the expression given by (5).

$$\frac{m_c}{m_c + m_f} \tag{4}$$

$$\frac{N_c.d_c}{N_c.d_c + N_f.d_f} \tag{5}$$

where m_c , m_f , N_c , d_c , N_f and d_f respectively represent the filler's mass, the fibre's mass, the number of filler voxels, the filler density (2900 kg.m⁻³), the number of fibre voxels and the fibre density (1500 kg.m⁻³).

Table 2 summarises the results obtained after processing the data described above.

Sample name	Added amount	X-Ray microtomography
Hard00	_	_
Hard05_1	5%	4.4%
Hard05_2	5%	4.6%
Hard10_1	10%	9.3%
Hard10_2	10%	9.2%
Hard20_1	20%	12.6%
Hard20_2	20%	12.9%

 Table 2
 Filler contents for hardwood samples

Table 2 shows that the same order of magnitude is found for each pair of samples. The rates calculated by the method increase as the known rates increase. The measured rates are underestimated which can be due to the digital aspects of the treatments. Namely the parameters used to separate the filler's phase from the other constituents have to be chosen. However, the high intensity value that appears corresponds to either phase contrast or to the filler's attenuation coefficient. Its smallest values have the same order of magnitude than the white fringe of phase contrast along the fibres' edges. Hence, a compromise has to be made.

For low filler contents, the amount of fillers found correspond approximately to the theoretical ones (the relative error is about 7%). The difference for high filler contents can be explained by the preparation of samples. In our case the samples were tilted and we had to remove a significant part of the sample from our analyses in order to evaluate the filler contents and not to take into account the "Post-it®" for filler evaluation. A commercial sample with known filler content was also imaged and processed with the same numerical treatments. As the preparation did not introduce a tilt, the estimation was carried out on the whole sample. The filler content of the analysed sample was estimated as 17.6% whereas the known global average was 19%. Therefore we may consider that the technique for evaluation of the filler content is appropriate.

4 APPLICATION: CHARACTERISATION OF STRUCTURAL PARAMETERS

4.1 Stereology measurements

The goal of this study is to characterise the fibre orientation and 3D structural parameters.

4.1.1 Samples' description

The samples analysed are three different oriented papers called 16, 19, and 20. They are made of bleached softwood fibres beaten to 25°SR. The different fibre orientations were obtained on an industrial machine changing the difference of velocities between the pulp jet and the wire on which the pulp is sent. These characteristics are summarised Table 3.

Table 3 Speed difference for oriented paper. The studied samples (Figure 11) are 700 \times 700 \times 20 voxels. Porosity study can be carried out since the studied volume is greater than the characteristic volume obtained, evaluating REV. The characteristic length is 170 μ m.

Paper	16	19	20
$V_{jet} - V_{wire}(m.min^{-1})$	30	-19	-5

4.1.2 Stereology measurements

Stereology is used to obtain information on 3D morphological properties of the studied material from 2D measurements. This technique is used to study slices of microtomographic data. For each slice, a parallel beam, that forms an angle θ with the abscissa axis, is used to compute the following quantities [22]:

• L_{θ} is the mean intercept number per unit of length in the direction θ between the beam and one interface.



Figure 11 Slice of the oriented papers from left to right: 16, 19 and 20.

• g_{θ} is the mean length in the porous phase on the whole space in the direction θ .

Let $\langle L_\theta\rangle$ and $\langle g_\theta\rangle$ be the average of these magnitudes evaluated for different angular positions.

4.1.3 Feasibility of the study

First, the magnitudes of the above presented parameters are evaluated in the three main directions (Table 4). Let

Sample	16	19	20
Lx	0,0567	0,0593	0,0646
Ly	0,0689	0,0764	0,0625
Lz	0,1517	0,1683	0,1685
Lmean	0,0924	0,1013	0,0985
gx	16,7871	11,2813	9,5034
gy	13,8227	8,7863	9,7225
gz	5,0200	3,0201	2,8453
Gmean(µm)	8,3136	7,6959	7,3571

 Table 4
 Measurements of intercept numbers and mean lengths.

• i be the directions x, y or z of the microtomograph,

- Li be the intercept number in the direction i,
- gi be the mean length in the direction i.

The rate $\frac{g_y \cdot L_y}{g_x \cdot L_x}$ is computed in order to compare it with its theoretical value equal to 1 (Table 5).

Sample	16	19	20
$(ly \times gy)/(lx \times gx)$	1,001	1,004	0,990

 Table 5
 Validation of the calculation of intercept numbers and mean lengths.

The calculated rate is close to 1 which validates the measurement techniques. Therefore we consider that the geometrical anisotropy can be studied.

4.1.4 Geometrical anisotropy

Geometrical anisotropy can be characterised by an ellipse and in particular by its ellipticity [23]. Namely a circle is characteristic of an isotropic structure whereas a higher ellipticity is the symbol of a higher anisotropy. Let a be the major axis of the ellipse and b the minor one. The rate a/b represents the ellipticity, which is classically used for anisotropy characterisation. These "standard" papers have been previously characterised using other tools [24]. Table 6 presents the ellipticities evaluated on microtomographic data and the ones presented in [24].

Sample	16	19	20
a/b (tomographic data)	1,24	1,33	1,1
a/b (results presented in [24])	1.7	1.8	1.4

 Table 6
 Geometrical anisotropy rate for oriented papers.

The obtained results are compatible to the values extracted from literature. Consequently, the geometrical anisotropy can be deduced from microtomographic data.

4.2 Granulometry

Granulometry deals with the study of the size distribution of the constituents [25]. For digital data, a classical morphological filter known as "opening" is applied. It consists of an erosion followed by a dilation with the same structural element. A granulometry is computed using a succession of openings of size λ . After applying each filter, the volume M_{λ} is measured. The curve $F_{\lambda} = 1 - \frac{M_{\lambda}}{M_0}$ is the distributive function, where M_0 represents the original



Distribution of cumulative volume frequency

Figure 12 Granulometry of paper. The structuring element is an octahedron.

volume. This sequence of filtering is applied to the binarised data using a 3D structural element. A cubic structural element of size n means a cube of $((2xn)+1)^3$ voxels. Given the morphology of the pores, an octahedral structural element is chosen. This numerical technique is applied to the bulk of the sample. Figure 12 is an example of granulometry obtained in the bulk of three different paper samples: a paper board, the handsheet 16 (softwood) and the handsheet hard00 (hardwood).

The granulometry curve gives access to the pore size repartition and gives information on the characteristics of the structure.

5 CONCLUSION

This paper deals with the characterisation of paper samples imaged with X-Ray synchrotron Radiation microtomography. X-Ray synchrotron radiation microtomography coupled with appropriate image processing tools is a good way to obtain quantitative parameters that characterise the 3D structure of papers. The first step consists of the segmentation of the data into

different phases, namely, air, fibres and fillers. The amount of each component may be evaluated. This was validated for both the porosity and the filler content. Furthermore, structural parameters were calculated from the binarised volumes. The comparison with the published results validates the calculation.

This technique and the developed method may be applied to both industrial samples and other materials. They can be used for example to study the influence of each unit operations of the paper making process. Finally, the obtained structures may be used to estimate physical properties of papers, such as thermal conductivity or permeability.

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Transcription of Discussion

CHARACTERISATION OF THE 3D PAPER STRUCTURE WITH X-RAY SYNCHROTRON RADIATION MICROTOMOGRAPHY

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Christian Schmid Hewlett-Packard

First of all – a good job. I am curious to know what is the smallest size that can be resolved with this kind of technique? I mean X-rays are pretty small.

Sabine Rolland du Roscoat

Yes, the current resolution is $0.7 \,\mu m$ and on the ID19 beam line you can reach 0.28 μm , but we are not sure that the size is relevant for quantitative measurements.

William Sampson

Your ash work has all been done on calcium carbonate as far as I can make out. Can you image clay in filled papers or are there problems in resolving it?

Sabine Rolland du Roscoat

I do not know because we have not tried and that was not the main focus of this study.

Discussion

William Sampson

No, I understand that. Thank you.

Jose Iribarne Solvay Paperboard LLC

Excellent work. I see that you have used this technique to characterize porosity and filler content but I did not see looking at fibres listed in your future work. I am wondering if anybody is planning on doing fibre distributions such as length, curl, kinks and so on. I would be really interested to know if what we measure in the liquid phase applies once the fibres are in the paper.

Sabine Rolland du Roscoat

First, you have to separate the fibres and it is not so easy. There is a Norwegian team starting to work on this.

Patrice Mangin U.Q.T.R./CIPP

You have shown us two slides: one about the distribution of the filler size and another one about distribution of fillers over depth. Do you have the availability in your data set to actually see the size distribution of fillers according to depth, it would be of very high interest to model fillers in sheet structures?

Sabine Rolland du Roscoat

Yes.

Thad Maloney KCL

Back to the fillers, of course this is a very valuable tool you have for the analysis of filler distribution and as just pointed out filler distribution through the depth, but could you comment a little bit? You seem to have some difficulty measuring the filler contents in the highly-filled papers and hand-sheets, but then you presented some industrial sheets and for example décor paper, 37% filler content, and there you seem to succeed, so, what is actually the difference?

Sabine Rolland du Roscoat

It was not done during the same acquisition campaign. So the capillaries were

not the same and the samples were not tilted. When the sample is tilted, we get some ring artifacts and they are detected as fibres. Consequently, it decreased the amount of the filler content. Actually we now use new capillaries and the samples are quite flat. Therefore, we do not have this problem anymore. Moreover, we have not measured the ash rate directly on the imaged handsheet. The handsheet prepared may have differed a little bit from the one used to evaluate the filler content. Furthermore, as demonstrated in the manuscript, the amounts of fillers were correctly evaluated for the industrial samples.

Thad Maloney

Just a follow up question. I suppose in the future, you will also make some more analysis on the filler flocculation within the sheets, the degree of agglomeration and so on?

Sabine Rolland du Roscoat

Yes.

William Sampson

You showed some data for the structural anisotropy that you have measured from your images and compared it with anisotropy measured using laser techniques and in each case the structural anisotropy was less than that you observed with lasers. We see the opposite effect when we look at the distribution of mass in the sheet. The sheet always appears more uniform in the plane than the distribution of mass and I wonder, is it always the case that structural anisotropy measured with light will seem to be greater than it actually is in the network? Is that your observation or have you looked at these three only?

Sabine Rolland du Roscoat

Yes, it has always been the case.

Jean-Claude Roux EFPG-INPG

Wonderful work. You characterized the characteristic lengths for porosity, however, if you consider physical properties, perhaps you would have other characteristic lengths? Can you comment on this?

Sabine Rolland du Roscoat

This work was started by Maxime Decain (PhD) and it has been evaluated for permeability and it may even be found that if you evaluate porosity for different volumes, you can estimate the mean permeability, and you get the same results for small volumes as for the huge volumes.



The above figure shows the permeability in the z direction and the results we found for several different parts of the sample (E1 to E5). On the right the volumes are huge and on the left the volumes are small. The mean of those values is computed for each volume (dashed line without data points, grey on colour image) and it is very similar to the mean for the whole sample volume (drawn as a dashed and dotted line, red on colour image).

Marit Van Lieshout Paperlinx

I was wondering, can you differentiate between the interfibre void areas and the intrafibre void areas, or in other words, the porosity that is located between the fibres and within the fibres?

Sabine Rolland du Roscoat

I do not think that we have sufficient resolution.