# Influence of paper structure on printability: Characterisation of paper using X-Ray Synchrotron Microtomography

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## Abstract

*Most of the end use properties of paper do depend on its structure. For example, considering printing, the efficiency is essentially related to its superficial structure. Therefore, the presentation will focus on a new tool used to visualise and characterise the fibrous structure.*

*The focus is set on wood fibre-based materials and the characterisation of such fibrous structures at microscopic level. An important aim of this research is to establish a relationship between the structure of papers and their physical properties. Characterisation of fibrous structures of wood fibre-based products is obviously a key factor for the development of the printing industry and also for the improvement of the final product. Therefore in order to optimise the printability, it is necessary to measure and to characterise the resulting structure at the fibrous level. For the wood fibre-based products, the characteristic size has an order of magnitude of few microns. First, some measurements were carried out in order to characterise the topography. of the papers. In a second part, some complementary measurements were carried out on the considered samples, using synchrotron source based X-Ray microtomography tools to describe the structure. Consequently, a 3D description of the fine fibrous structure is obtained from X-Ray microtomography. The experimental set-up will be briefly presented. We have carried out experiments at the European Synchrotron Radiation Facility (ESRF) located at Grenoble. The presented examples will illustrate the potential of this technique to describe such structures and therefore optimise the final product.*

## Introduction

The increasing demand for high quality printing has lead to focus the research effort on the paper support. Indeed, it may modify the quality due essentially to its superficial properties and the wetting time, for example. Hence, many efforts were carried out to characterise its topography. We would briefly present some results concerning this 2D characterisation showing that some interesting results may be obtained, an in particular, the two-sidedness effect may be foresee. Indeed, a simple characterisation of the surface (using the parameter Sq) proves that both sides are different. In the second part, the 3D characterisation will be presented. First we will describe briefly the used tool (X-Ray microtomography). Then, the presented results will prove the efficiency of such technique. Finally we will conclude this presentation essentially introducing perspectives that may lead to the improvement of printing.

## 1 – Surface analysis: Topography

- In order to characterise the surface, classical tool may be used. For example, the following images (Fig.1 and 2) have been obtained using a Scanning Electron Microscope. Two different papers, which differ in a printing quality point of view, are imaged. One may observe the different surface structures of both papers.

Fig. 1: Scanning electron microscopy: Paper 1 (scale: 66.7 and 16.7 microns, respectively)

Fig. 2: Scanning electron microscopy: paper 2 (scale: 66.7 and 16.7 microns, respectively)

Elementary analysis may be carried out on these surfaces.

- Some other techniques may be useful to characterise the surface topography. To illustrate this possibility, we analysed different papers dedicated to digital printing, using an optical profilometer (Altimet[@]): Paper 1 to 4 (Fig. 3). The measurement conditions were: - for X and Y axis: 6 mm, 1500 points (leading to a 4 micron spacing), - for the Z axis resolution: 9 nm resolution.

Fig. 3: Optical profilometry: two sidedness effect (Paper 1 to 4, resp. side a and b)

The obtained results confirm the claimed differences of quality for the different commercial papers. Indeed, first, the visual aspects are different from a paper to another, concerning either both sides or the comparison of two sides. Secondly, to confirm, this visual observation the standard parameter Sq was used as an indicator of quality. The results are presented in Tab. 1.

| Sq (µm) / Paper | 1 | 2 | 3 | 4 |
|---|---|---|---|---|
| Side A | 9.97 | 4.98 | 6.57 | 6.42 |
| Side B | 8.31 | 4.62 | 0.96 | 1.05 |

Table 1: Values of the surface parameter Sq, for the 4 different papers for each side.

Theses results may in particular cases discriminate the different paper qualities. However, considering ink penetration, we have to consider the 3D structure of the fibrous material, that is to say paper. Consequently, we will present now the experiments carried out to characterise the 3D structure of papers.

## 2 – Volume analysis: Synchrotron X-Ray microtomography

The work presented in this paper was carried out on microtomographies of various paper samples acquired by X-Ray Synchrotron Radiation microtomography [1,2]. This technique gives access to the 3D structure of the samples in a non destructive way. All the data presented in this paper were acquired on the ID19 beam line of the European Synchrotron Radiation Facility, located in France. The used equipment is schematically depicted in Fig. 4. The main goal of microtomography is to build a 3D representation of the inner structure of a sample [3]. During data acquisitions, a monochromatic beam (20.5 keV) irradiates the sample that is

placed on a rotation stage. The transmitted beam is recorded for 1500 angular positions between 0 and 180 degrees. A filtered back projection algorithm is applied to the set of the radiographs to reconstruct the 3D structure. The data obtained represents a 3D map of the absorption coefficient of the sample structure.

Fig. 4: Schematic view of the data acquisition involved at the ESRF installation

An example of the obtained 3D structure is presented in Fig. 5. We may observe the 3D structure. It is important to note that the local structure is known at any point of the structure. We may therefore extract cross sections in any direction. Moreover, to be able to analyse quantitatively the structure, segmentation was carried out [4]. The same cross section has been extracted from the obtained segmented structure. The superposition of both sections validates our image analysis.

Fig. 5: 3D view of a paper, a grey level cross section and its segmented visualisation

The method was applied to the studied papers. The results are illustrated in Fig. 6 to 8. In sake of simplicity we represent here paper 1 to 3, as the visual conclusion are obvious. Moreover we may, in some cases, observe the coating layer. One has to remember that the chosen resolution was 0.7 micron. Consequently, using a (2048x2048) camera, the visualisation concerns a total length of 1.4 mm.

Fig. 6: Paper 1 (3D (981 µm * 981 µm) and cross section (371 µm * 70 µm) views)

Fig. 7: Paper 1 (in plane view: Grey levels and segmented images)

Fig. 8: Paper 3 (in plane and cross views)

We may, for example, observe in Fig. 8 the two-sidedness effect, already mentioned during the analysis of the results in Tab. 1. Furthermore, the coating layer is clearly observed;

Then new descriptions may be proposed. As an illustration, we present in Fig. 9, the porosity profile in the thickness direction. We insist on the point that the porosity is evaluated for each position on the whole horizontal points. Using such analyse, two conclusions may be directly drawn. First, paper presents a bulk and surface effects due to the making process. Moreover, we may observe in some cases, differences in the surface porosity gradient, which signifies a two-sidedness effect.

Fig. 9: Porosity profile (thickness direction) for paper 1

Finally, we would like to point out, other possibility of such technique. As we were able to separate the pores and the fibres, we were also able to distinguish between fibres and fillers [5] . Using our developed software, we may extract from the paper measurement the different positions of fillers, as depicted in Fig. 10.

Fig. 10: 3D view of the fibrous structure and fillers localisation within the structure

Consequently, we are able to visualise and quantify the filler content. Moreover, a granulometry of the fillers may also be evaluated, which complete the filler localisation within the paper sheet. These characterisations are of most importance considering the paper quality in term of printing. Indeed, both the optical properties and the filtration process of inks are directly linked to theses factors.

## 3 - Conclusion

The proposed characterisation allows analysing in details the 3D structure of paper at a fine scale (submicron scale). Data may be extracted directly from the measurement, such as the porosity for example. Moreover, the gradient of porosity is clearly observed. It may, for example, indicate a probable two-sidedness effect. Furthermore, the fine description of the fibrous media is necessary in order to better understand the relationship between ink and paper. Consequently, the optimisation of the considered paper-printer may be investigated in some future works.

## References

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[5] Roscoat, S.R.d., J.-F. Bloch, and X. Thibault. Characterisation of the 3D paper structure with X-Ray Synchrotron Radiation microtomography. in 13th Fundamental Research Symposium. 2005. Cambridge: The pulp and paper Fundamental Research society. Pages

**Author Biography**

J.-F. Bloch: Associate Prof. at EFPG. Specialist of Paper physics. The author is a specialist fibrous structure and their related physical properties. He has organised International Conferences in Papermaking Science. He has been invited to few PhD defences both in France and abroad in the domain of paper physics and simulation of unit operations involved in the paper making process. He is involved in both academic and industrial projects of research.

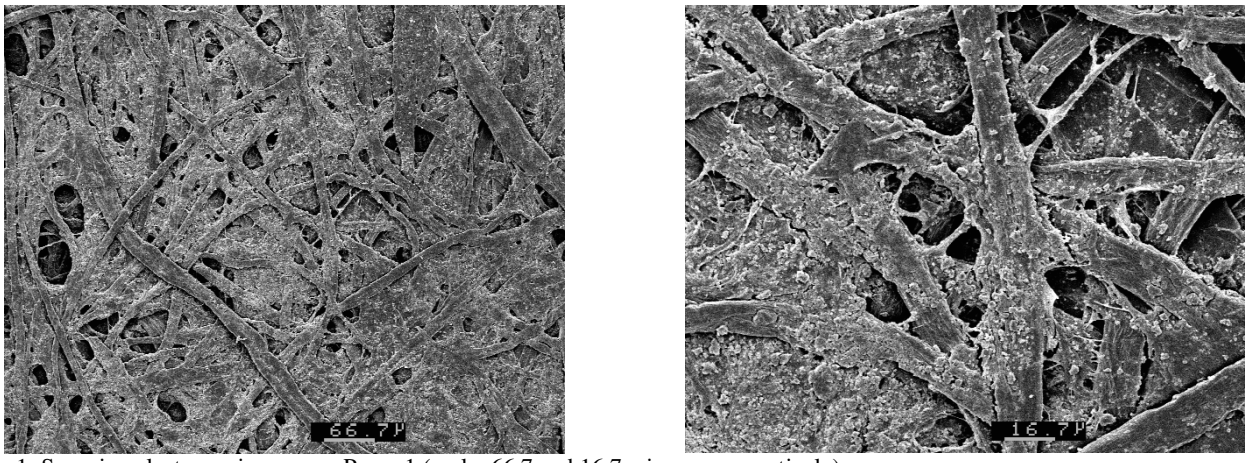



Fig. 1: Scanning electron microscopy: Paper 1 (scale: 66.7 and 16.7 microns, respectively)

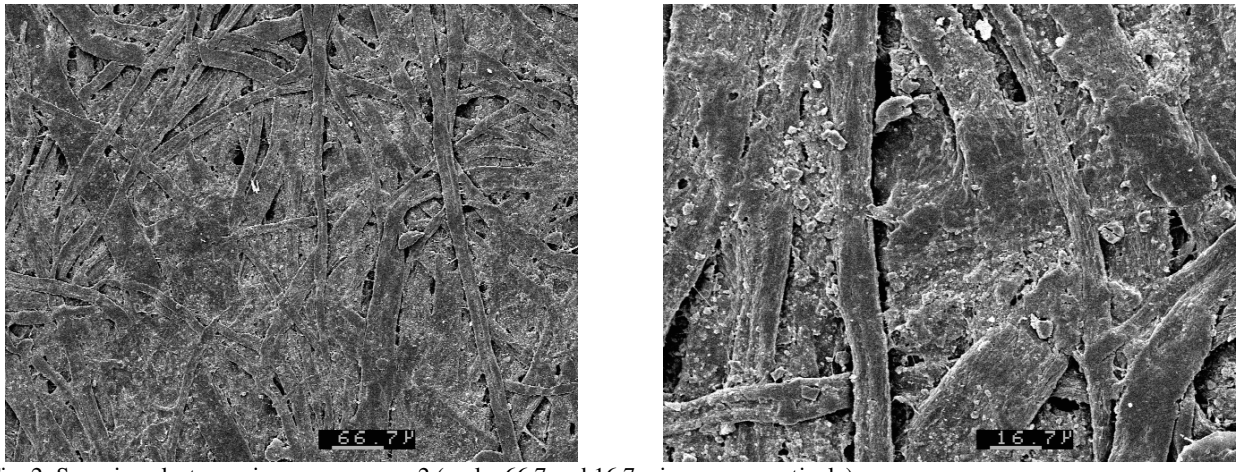
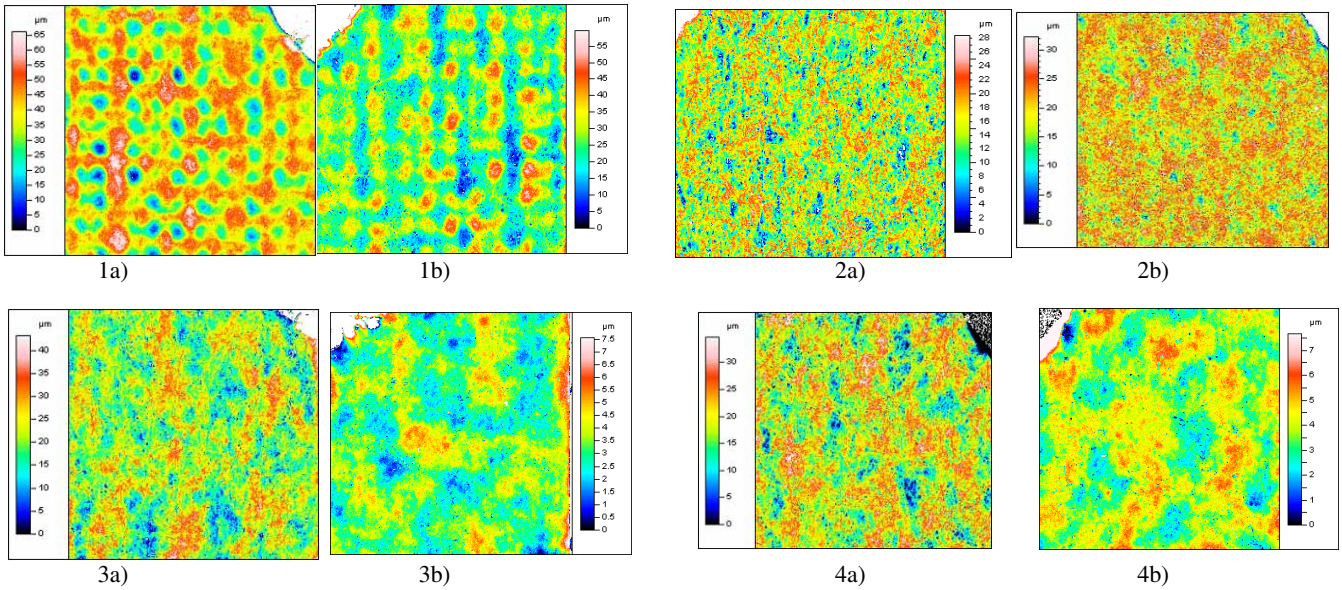



Fig. 2: Scanning electron microscopy: paper 2 (scale: 66.7 and 16.7 microns, respectively)



1a) 1b) 2a) 2b)

3a) 3b) 4a) 4b)

Fig. 3: Optical profilometry: two sidedness effect (Paper 1 to 4, resp. side a and b) – Measurements on 6x6 mm²

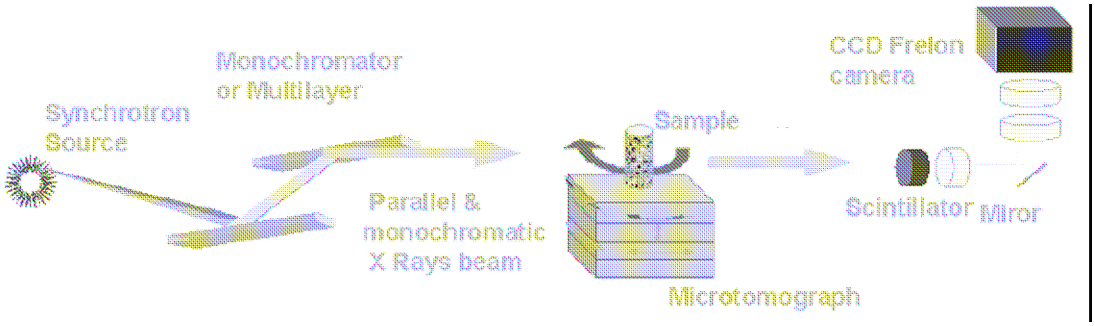
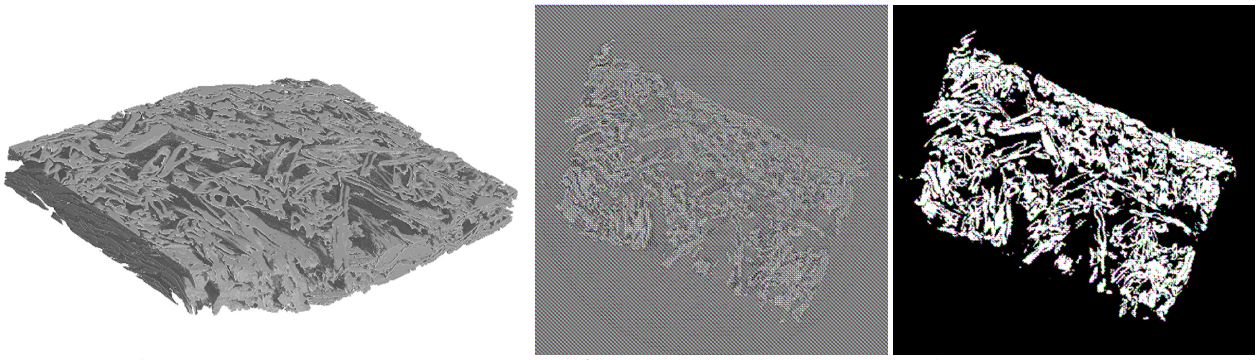



Fig. 4: Schematic view of the data acquisition involved at the ESRF installation



a) b) c)

Fig. 5: 3D view of a paper (a), a grey level cross section (b) and its segmented visualisation (c)

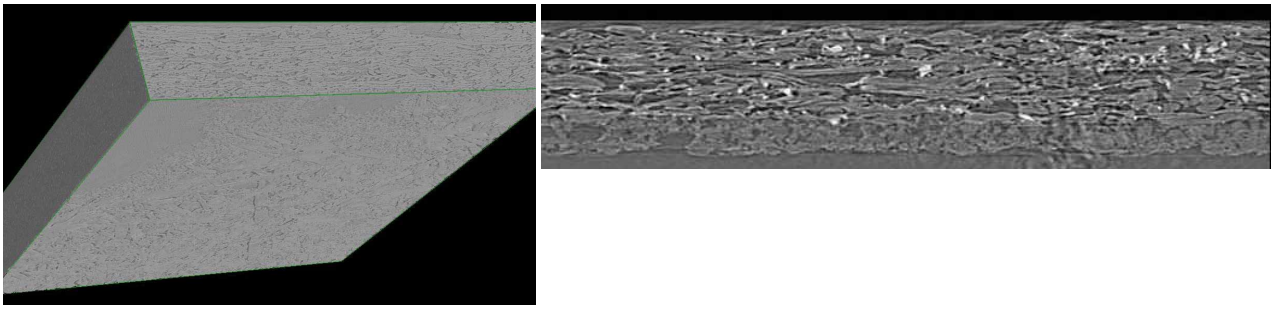


Fig. 6: Paper 1 (3D (981 µm * 981 µm) and cross section (371 µm * 70 µm) views)

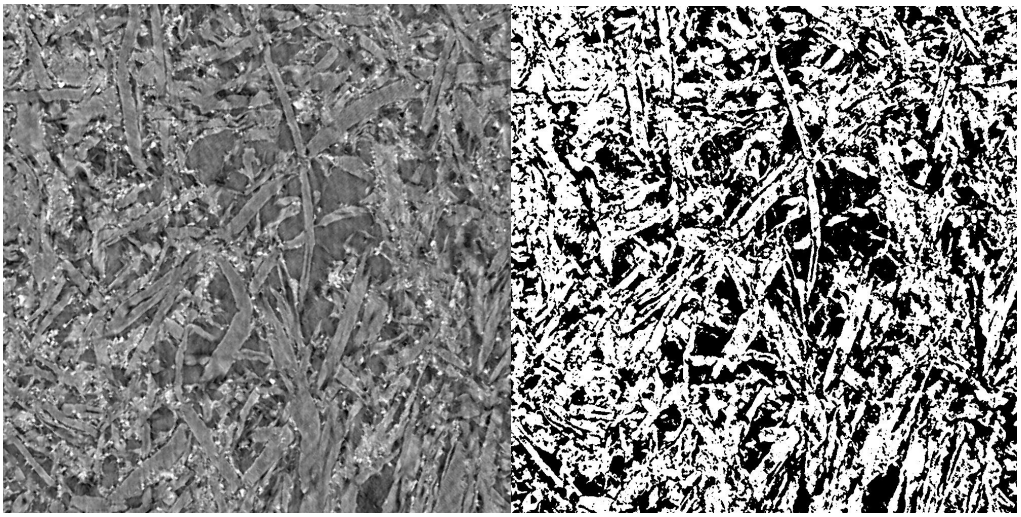


Fig. 7: Paper 1 (in plane view: Grey levels and segmented images)

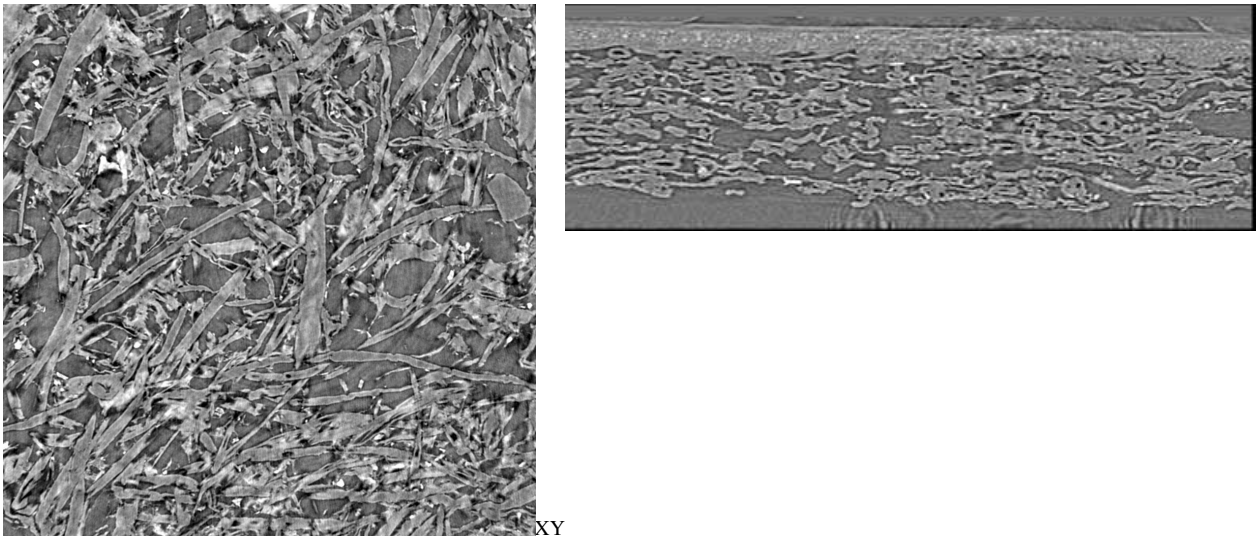

XY

Fig. 8: Paper 3 (in plane (981 µm * 981 µm) and (292 µm * 95.9 µm) cross views)

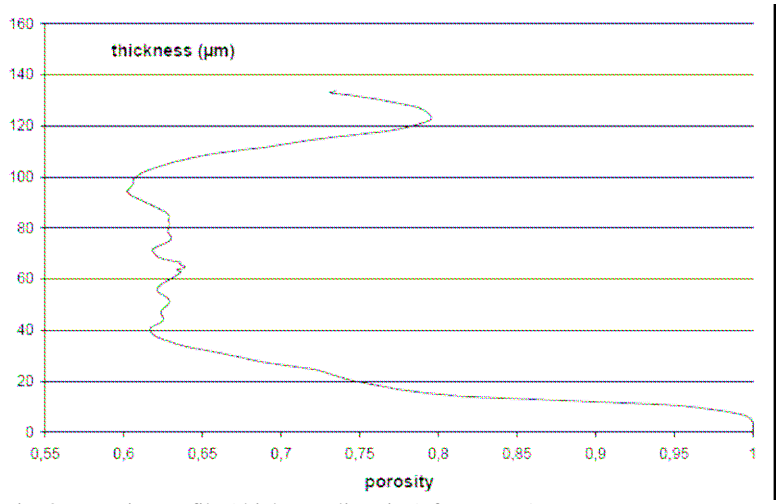



Fig. 9: Porosity profile (thickness direction) for paper 1

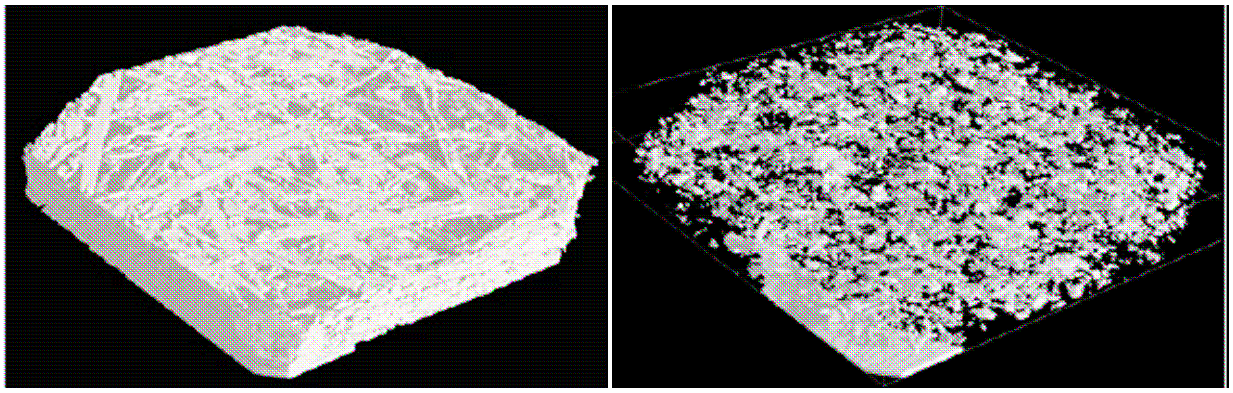


Fig. 10: 3D view of the fibrous structure and fillers localisation within the structure.