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Report:

Introduction:

This long term project was dedicated to the investigation of wood fibre based materials, especially to papers at a microscopic scale. An important aim of this project was to establish a relationship between the structure and its physical properties. This report summarises the experimental protocol, the numerical developments and the obtained results.

Materials

- Samples:

As wood fibre based materials cover a large area from a structural point of view, different kinds of samples were visualised. The analysed samples can be divided into two main categories. The first one consists of handsheets samples. They were prepared in laboratories to see the effect of unit operations of the paper production such as effect of pressing, refining, and the influence of the raw material. They were prepared by the French and US teams. The second one concerns paper samples prepared on pilot machines. Those samples were chosen in order to study the link between structure properties and end-use properties such as printing paper (French, Finland), food-board (US, Finland), fluff fibres samples (Norway), composites reinforced with wood-fibres (Norway).

- Sample environments:

The wood fibre based materials are submitted to different sorts of constraints during their use. That is why different compression devices were developed and implemented on the microtomograph (France and Finland). These compression devices were first applied to blotting paper for testing and then to layered cardboard and printing paper.

Paper is a material that is sensitive to its environmental conditions. That is why two different techniques to apply different levels of humidity, or to control humidity, during scans have been developed and tested (France). These samples' environments were applied to several paper samples: the structure evolution for different making parameters and the behaviours of paper having different end-use properties. Two third of the analysed samples were papers of different grades.

Other samples studied were composites where the influence of fibres on the structure was studied.

Methods developed:

- Data analysis technique:

Data obtained by X-Ray Synchrotron Radiation microtomography are in grey levels. To analyse the structure, segmentation has to be performed. Different tools to binaries the data have been developed by each team and compared. Fast methods were developed by Norway, Finland and US teams. They are based on filtering and adapted thresholds. However, those methods do not allow to separate a three phase paper (pores, fibres and fillers, for example). That is why other tools dedicated to paper samples were developed and applied to paper samples (France).

- Data simulation

3D algorithms to simulate moisture transport and permeability were developed and/or improved and applied to samples acquired during this project.

Results:

- Experimental part.

The first aim of the work was to find the experimental protocol for paper and board analysis and to validate it. Therefore, the first step consisted of an optimisation of the beamline parameters to the specific studied samples in terms of signal to noise ratio and data acquisition time (France). To image the large field of samples analysed, improvements in the sample preparation and in holders have been proposed (Norway, France) and tested during each run. To validate the repeatability of the experimental procedure, samples were extracted from the same original materials and we verified that the measured parameters were similar (France).

The second part of this project was to develop dedicated sample environments. Compression devices were designed, tested and successfully applied on different paper samples (Finland and France). The scans performed for different compression levels allow to gain understanding on the behaviour of the structure in some important industrial processes such as wet pressing of paper. As the studied materials are sensitive to their environment especially to temperature and humidity conditions, humidity controlled scans were performed. The initial idea was to enclose the sample in a small cylinder where we controlled the humidity with "salt methods". Unfortunately, this method did not give the expected results. This could be due to two different factors. As the device was built to be easily used to change samples and humidity degree, the box was closed by screws. It was maybe not perfectly water proof. On the other hand, the device was built in Plexiglas which can also absorb humidity. Therefore a second sample environment was tested where the humidity level around the samples was controlled by a wet air generator. This last set-up appeared more adapted and gave good results. Samples were submitted to humidity cycles: 50%, 20%, 80%, 20%, and 50% Relative Humidity. Differences can be noticed on the obtained radiographs.

- Structural analysis

Structural analysis has been carried out on the pore phase on the binarised data (France) such as: porosity evaluation, stereology measurements, granulometry on the pore phase. Obtained results on microtomographic data were compared to those obtained with traditional measurements which validate the developed methods. The small volume imaged (compared to a sheet of paper) has been proved to be representative from a porosity point of view.

Quantitative measurements were done on microtomographic data (France). Filler contents were evaluated both on handsheet samples whose amount of added fillers was known and on industrial papers. The amount estimated on microtomographic data corresponds to the one added or to the ash rates. The small volume imaged is therefore representative from a filler point of view.

The influence of unit process step was studied (Norway, US). The influence of fractures was also characterised (Norway).

Once the data were segmented, different algorithms were applied to individualise the fibres. Methods were developed to separate the different fibres (Norway). They were also applied to composites materials (Norway).

- Simulations.

Many paper properties depend on transverse permeability. It is linked to the pore connectivity and pore size distribution in the thickness direction. Lattice-Boltzmann simulations were successfully applied to paper samples acquired on ID19 (Finland, US). The simulations carried out on paper samples show that the volume imaged is representative from a permeability point of view (France). Diffusive moisture transport in paper structure was also studied (Finland , US). The results indicate that the methods used can indeed be used to gain valuable information e.g. for modeling industrial processes such as wet pressing, drying and printing of paper.

Conclusion & perspectives:

This long-term project was the first extensive international joint effort in utilizing high-resolution x-ray tomography in wood-based materials reseach. A vast amount of data of excellent quality was collected during the project. Although only part of this bulk of data has been fully analyzed and published at the time of this writing, the project has proven successful and will eventually yield a number of additional publications and valuable results with applications in industrial problems. Moreover, the results already reported in international conferences are likely to promote the use of x-ray tomographic techniques as a general tool for materials structure analysis within paper-making industry and the related research community.

Reviewed publications:

1. Holmstad, R., C. Antoine, et al. (2003). "Quantification of the three-dimensional paper structure: Methods and Potential." Pulp and Paper Canda **104**(7): 47-50.
2. Ramaswamy, S., M. Gupta, et al. (2004). "The 3D structure of fabric and its relationship to liquid and vapour transport." Colloids and Surfaces A: Physicochemical and Engineering Aspects **241**: 323-333.
3. Holmstad, R., U. Aaltosalmi, et al. (2005). "Comparison of 3D structural characteristics of high and low resolution x-ray microtromographic images of paper." Nordic Pulp and Paper Research Journal **20**(3).
4. Hyväluoma, J., P. Raiskinmäki, et al. (2005). "Simulation of liquid penetration in paper." Physical Reviews E.
5. Roscoat, S. R. d., J.-F. Bloch, et al. (2005). "Synchrotron Radiation microtomography applied to paper investigation." Journal of Physics D: Applied Physics.
6. Thibault, X., S. R. d. Roscoat, et al. (2005). "Experimental improvement for microtomography of paper and board." Computational Methods and Experiments in Material Characterisation II: pp 207-216.

Conference proceedings and contributions:

1. Aaltosalmi, U., M. Kataja, et al. (2003). Numerical analysis of fluid and flow through fibrous materials. International Paper Physics Conference, Victoria, BC, Canada.
2. Gupta, M., A. Goel, et al. (2003). Analysis of z-directional 3D structural characteristics of commercial paper and hand sheets. International Paper Physics Conference, Victoria, British Columbia, Canada.
3. Holmstad, R., S. Ramaswamy, et al. (2003). Comparison of 3D structural characteristics of high and low resoltuion X-Ray imaging microtomogrpahic images and paper and board. International Paper Physics Conference, Victoria, British Columbia, Canada.
4. Hyväluoma, J., P. Raiskinmäki, et al. (2003). Modelling the intrusion of non-wetting liquid into paper. Workshop on modelling and imaging of fibrous structures, Jyväskylä.
5. Ramaswamy, S., M. Gupta, et al. (2003). The 3D structure of fabric and its relation to liquid and vapor transport, characterisation of porous materials from angstrom to millimeters. Thrid International TRI/Princeton Workshop.
6. Gregersen, O. W. and R. Holmstad (2004). Fibre damage during mechanical pulping and calendering. The 2004 Progress in Paper Physics Seminar, Trondheim,Norway.
7. Hagen, M., R. Holen, et al. (2004). Digital Identification of connected paper fibres with cracks. The 2004 Progress in Paper Physics Seminar, Trondheim, Norway
8. Holmstad, R., A. Goel, et al. (2004). Effect of papermaking variables on the detailed 3D paper structure assessed by X-Ray Microtomography. The 2004 Progress in Paper Physics Seminar, Trondheim, Norway.
9. Hyväluoma, J., P. Raiskinmäki, et al. (2004). Intrusion of non-wetting liquid in paper. XXXVIII Annual conference of the Finnish Physical Society, Oulu, Finland.
10. Knackstedt, M. A., C. H. Arns, et al. (2004). Characterisation of 3D paper structure and transport properties of paper from tomographic images. The 2004 Progress in Paper Physics Seminar, Trondheim, Norway.
11. Roscoat, S. R. d., J.-F. Bloch, et al. (2004). X-ray microtomography applied to paper investigation. 7th Biennal Conference on High Resolution X-Ray Diffraction and Imaging, Prague.
12. Roscoat, S. R. d., X. Thibault, et al. (2004). Image analysis of 3D structure : Application to X-Ray microtomography. The 2004 Progress in Paper Physics Seminar, Trondheim, Norway.
13. Wang, L., S. Ramaswamy, et al. (2004). Surface Structure charcterization of porous media using x-ray micro computed tomogrpahy. The 2004 Progress in Paper Physics Seminar, Trondheim, Norway.
14. Roscoat, S. R. d., J.-F. Bloch, et al. (2005). Characterisation of the 3D paper structure with X-Ray Synchrotron Radiation microtomography. 13th Fundamental Research Symposium, Cambridge, The pulp and paper Fundamental Research society.
15. Turpeinen, T. (2005). Computerized tomogrpahic imaging of paper coating layers. the 22nd coating symposium, Munich, Germany.

Theses:

1. Aaltosalmi, U. Fluid flow in porous media with the lattice-Boltzmann method. Department of Physics, University of Jyväskylä, Jyväskylä, Finland, July 2005.
2. Holmstad, R. Methods for paper structure characterisation by means of image analysis, Norwegian University of Science and Technology, Norway, July 2004.