ESRF	**Experiment title:** 3D –imaging of fibrous filters through high resolution X ray tomography	**Experiment number:** ME-924
Beamline:	**Date of experiment:** from: September 9, 2004 to: September 11, 2004	**Date of report:** March 14, 2005
Shifts:	**Local contact(s):** Xavier Thibault	*Received at ESRF*

Names and affiliations of applicants (* indicates experimentalists):

Martine MEIRELES* (Laboratoire de Génie Chimique, Toulouse)

Jean Christophe REMIGY * (Laboratoire de Génie Chimique, Toulouse)

Mehdi REBAI * (Institut de Mécanique des Fluides, Toulouse)

1.Aim of the experiment

Filtration of fine particles through fibrous filters involves the capture of particles within the three-dimensional network formed by the fibers entanglement. Several physical mechanisms that govern particle capture in the vicinity of a fiber (direct interception, inertia, sieving, diffusion) have been described at the fiber particle micro-scale and modeled for simplified array configurations of infinite lengths. As it quite complex to describe all of these mechanisms in a single model, one has to choose which of these is (are) dominant. Today , the answer to this question is based on physical analysis of the ratio of fiber-particle interaction to transport phenomena (convection, diffusion) at fiber-micro scale level. This implies that collective particle transport as well as complex geometrical features of fiber entanglement are entirely neglected. As a consequence, although such theoretical approaches could be validated with experiments carried out for simple configurations, there is still a large gap between these models and the global macroscopical properties of fibrous filters. In practice, none of these approaches is actually involved in filter design which is essentially based on extensive and costly experimental campaigns.

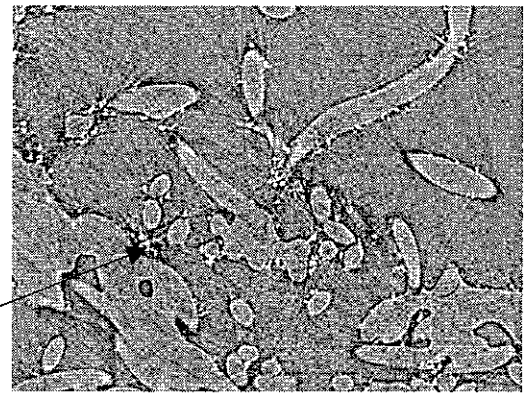
A multiscale description has been recently develop [1] to fill the gap between microscale level approaches and macroscopical parameters such as efficiency or lifetime. It is based on a modeling of particle sieving mechanism which includes the complexity of porous geometry through a detailed description of interconnected structure in terms of pores [2]. Data were acquired by ESEM. Yet such a detailed description is limited as based concepts of connectivity and tortuosity are also needed for "real geometrical features".

X ray tomography may be used for mapping in detail a fiber structure in three dimensional digital to resolve the complex geometrical features with micrometer resolution.

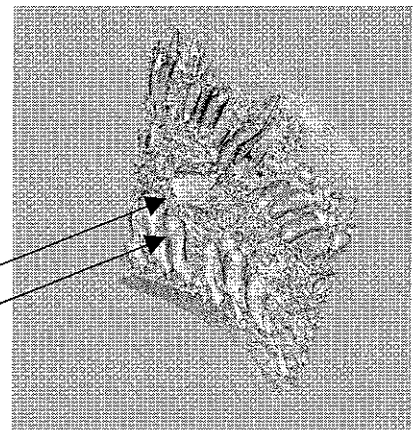
Whether the particle capture mechanism is a sieving mechanism that directly implies the geometrical structure of fibers or a surface mechanism (interception, adhesion) which depend on physico-chemical interactions in the vicinity of the fiber is also a point of debate which could not be solved by previous approaches. Indeed, the ratio which defines two distinct regimes between "surface" mechanisms and "flow mechanism" is again only define for simple 2D array configuration. X ray tomography may then be used for mapping the localization of particle within a clogged filter as one expects that when a "surface" mechanism

dominates particles are captured on fiber length whereas for the other mechanism they are stopped by fibers entanglement.

2. Results of the experiment



During ME-692 experiment, we imaged four different medias produced by several routes. We also investigate each at different level of clogging. We had some difficulties with volume reconstruction during the experiment. The reconstruction was carried out later on by X.Thibault and the data came out recently. Nevertheless, we had quite interesting images that we just started to treat. The first figure included here is an example of a clogged filter where particle captured by the fibers can be imaged (particles are about 2 microns, fibers are 2 mm).
This example is a case where one would expect a "surface" driven mechanism (particle are a tenth of the fiber), but imaging shows that some particles are also captured in between the fibers. This implies a lower flow than expected from a " surface" mechanism, and a shorter lifetime. Many more information are expected from the data sets collected during the experiment.



Besides fibrous structures, we also seek to images internal microstructure of microfiltration membranes also used for filtration of submicron particles. These medias are produced through a phase inversion process. Of course, the resolution is still too low to image the features of the active top layer (pore sizes are less than 1 μm), but we obtained very useful information both on the internal macrostructure which appears to be a stack of different layers and on the presence of large defects in the active layer (defects larger than 1 μm) which may explain uncertainty in the standard characterization method used to test the "cut-off", [3],[4].

3. Concluding remarks

This experimental run was a confirmation of the oustanding potentialities of high resolution X-Ray tomography for imaging porous structures.
Concerning fibrous filters, highly valuable information can be worked out from such a technique in terms of characterization of geometrical structure but also in terms of understanding the physics of transport in "real" complex geometry . Breakthrough in the design of the filters and the modeling of filtration are expected from such information. Quantitative analysis of the 3D images both for the characterization of the interconnected structure and the identification of particles within the media is certainly the next task to achieve. Cooperative exchange programs with two different groups (applied mathematicians) have been organized to test different methods and codes devoted to morphological analysis.
3D imaging of a membrane structure is a task never attempted before . Again highly valuable information can be gained through this technique and topics such as membrane characterization and membrane formation could be better addressed. A new proposal addressing both directions has been recently submitted.

[1] Etude et modélisation du colmatage d'un filtre plissé, K.Benmachou, thèse de Doctorat, INP, Toulouse, 2005.
[2] Dynamic clogging of a pleated filter element : experimental and and theoretical approaches for simulation, K.Benmachou, P ; Schmitz , M Meireles, Communication at Filtech europa, Dusseldorf, Oct 21-23, 2004, submission to Chem. Eng. Science
[3] Relation Structure/Propriétés de transfert dans les contacteurs membranaires : Application à la séparation de composés d'arôme, F. Gascons Viladomat[1], V. Athes[1], M. Marin[1], I Souchon[1]
J C. Remigy[2], J C. Rouch[2], M. Meireles[2] ,Colloque Prosetia, 15 mars 2005
[4] X-ray tomography: a tool for membrane engineering?, L. Quezada, JC Rouch, J C. Remigy, M Meireles International Congress On Membrane, Seoul, August, 2005, submission to Journal of Membrane Science.