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# NMR methods and devices for the characterization of flows and transfers in milli-channels

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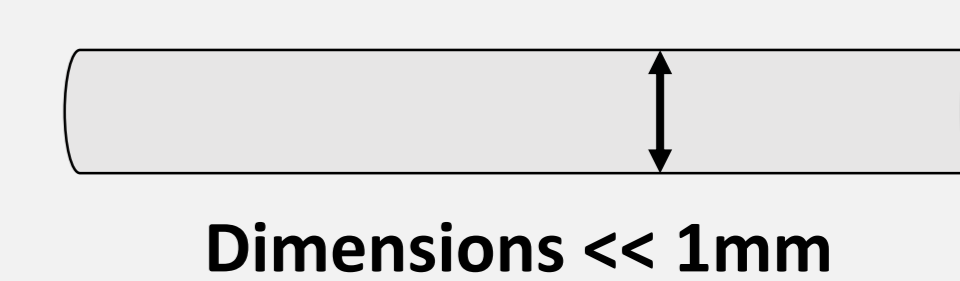
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## 1. Introduction

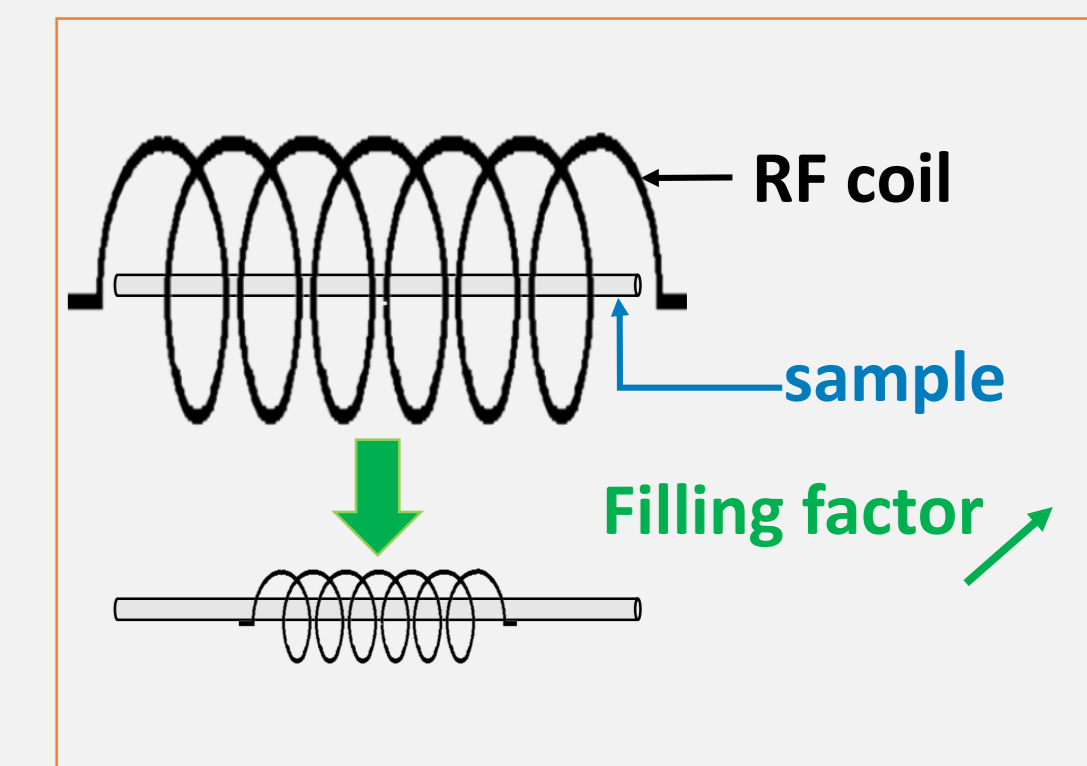
- Milli-fluidics is the technology of flows in channels with characteristic dimensions of a few hundred microns
- Reducing the dimensions of the systems affects the equilibrium of the forces and generates a specific behavior of the flow<sup>1</sup>
- Milli-fluidic systems are implemented in different fields such as synthesis chemistry, biology or process engineering<sup>2</sup>
- The geometrical complexity of the systems induces a limits the use of optical characterization methods
- NMR methods are adapted to such systems (geometries and materials)<sup>3</sup> provided that the measurement sensitivity is improved<sup>4,5</sup>



➤ **Approach: development of radiofrequency (RF) milli-coils<sup>6</sup> for emission and reception of the NMR signal**

**In this study**

- We developed adapted methods for the characterization of flows in milli-fluidic systems by NMR/MRI
  - Increase the sensitivity by developing RF coils
  - Optimize imaging parameters
- We applied these methods in two study cases:
  - Characterize the hydrodynamics in micro-mixer
  - Visualize a biofilm development in a capillary

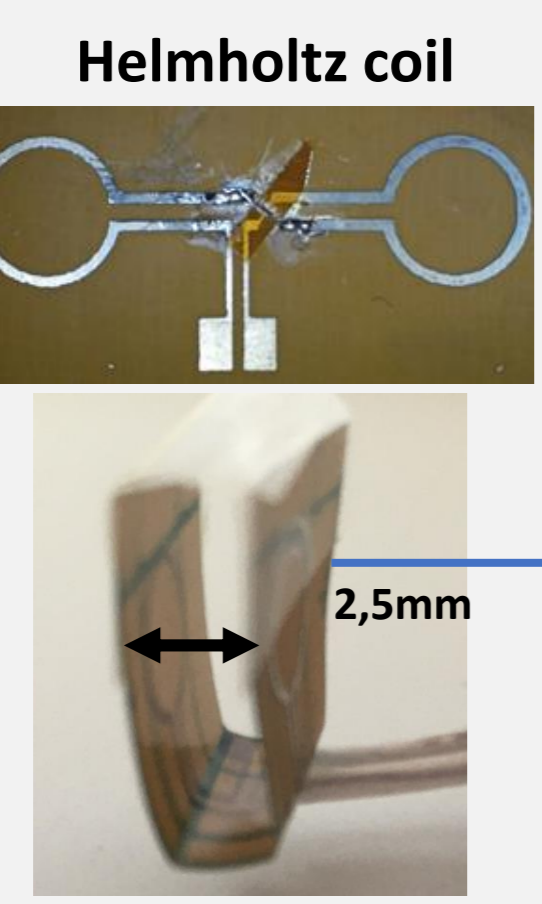
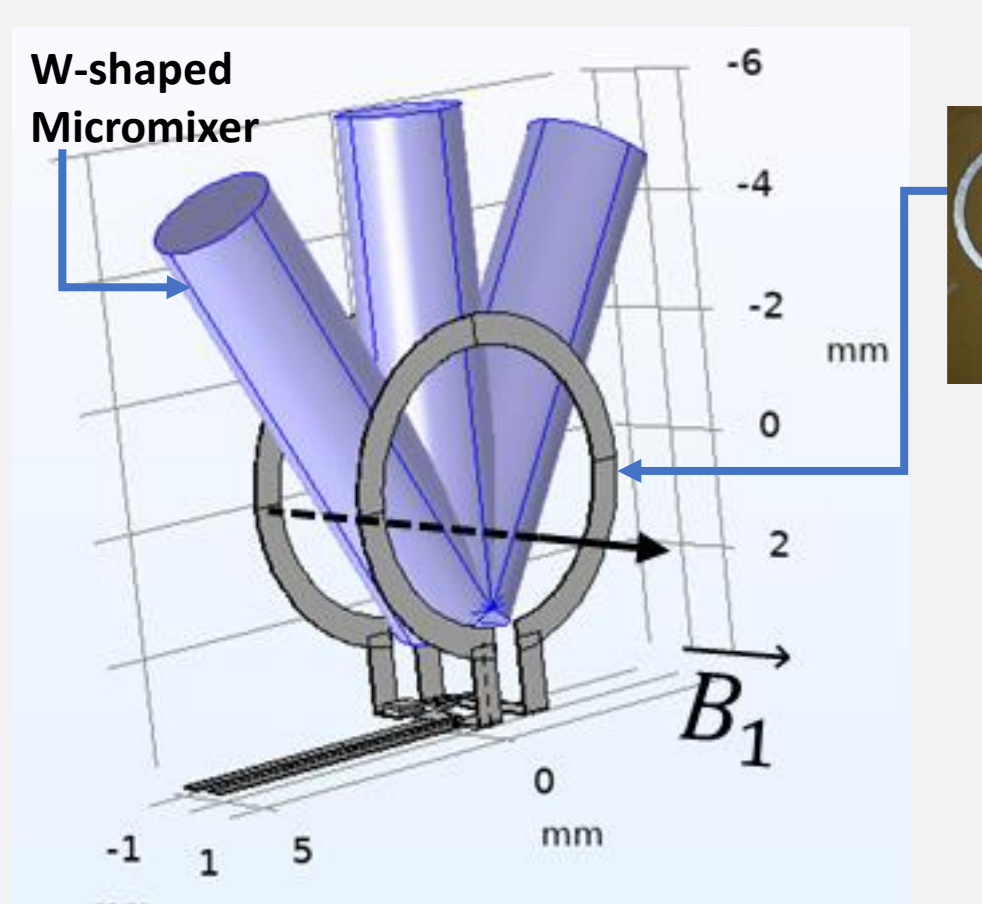


## 2. Materials and methods

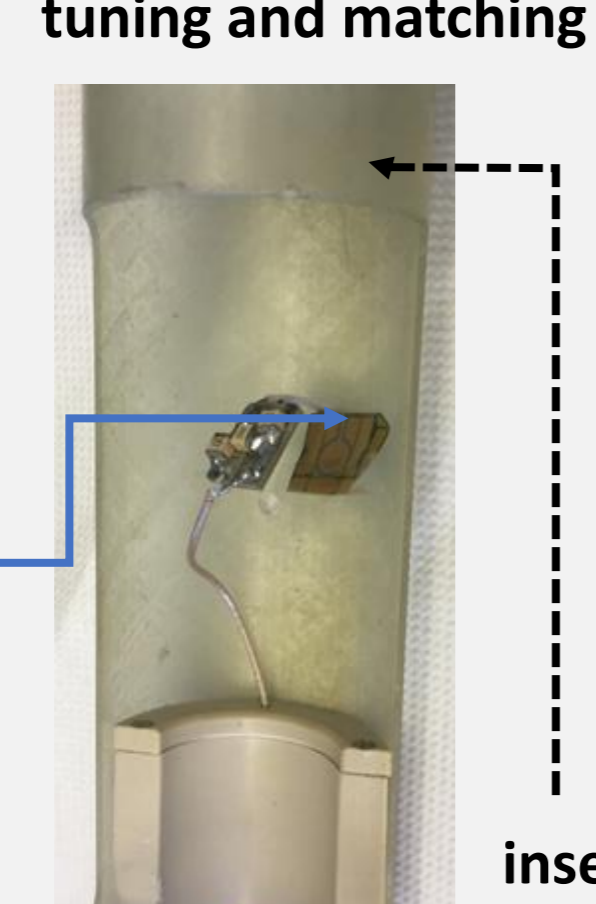
### • Fabrication of RF milli-coils

Etching on DuPont™ Pyralux® AC substrate

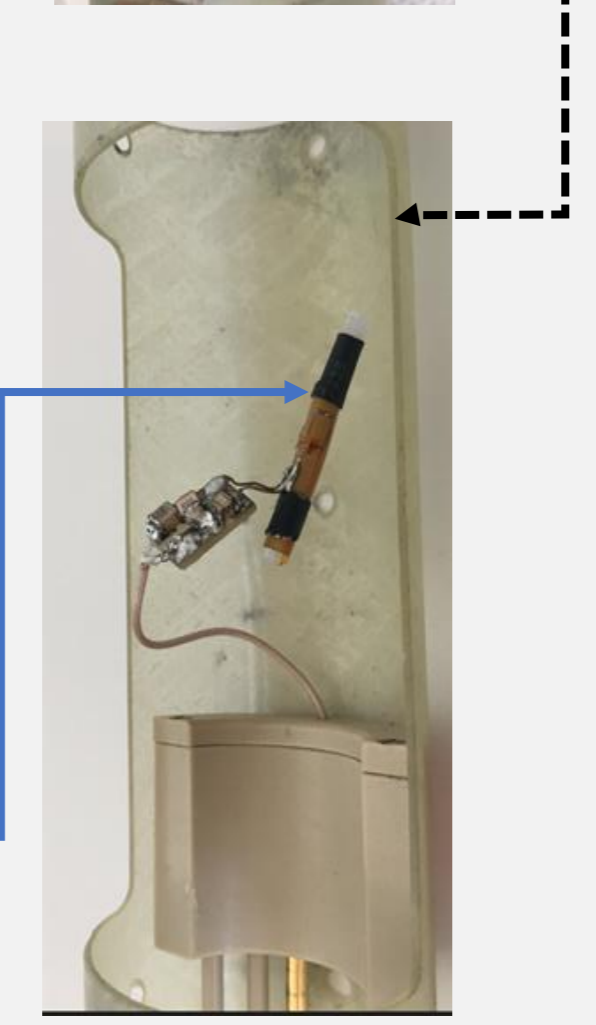
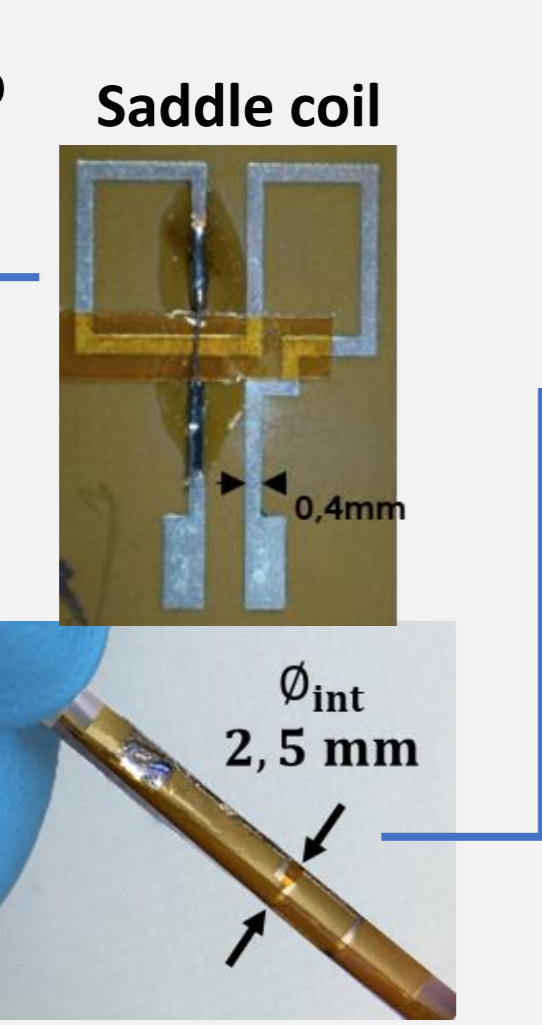
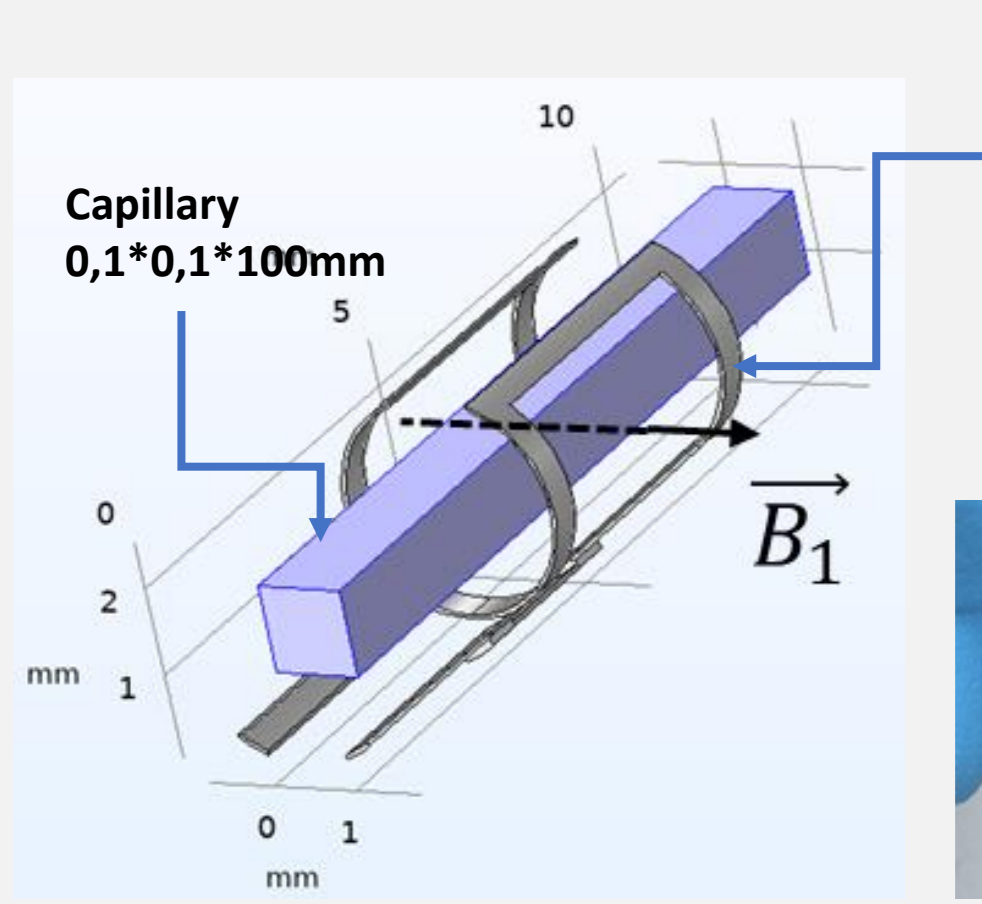
Chosen geometry for application 3.a



Electronics: tuning and matching

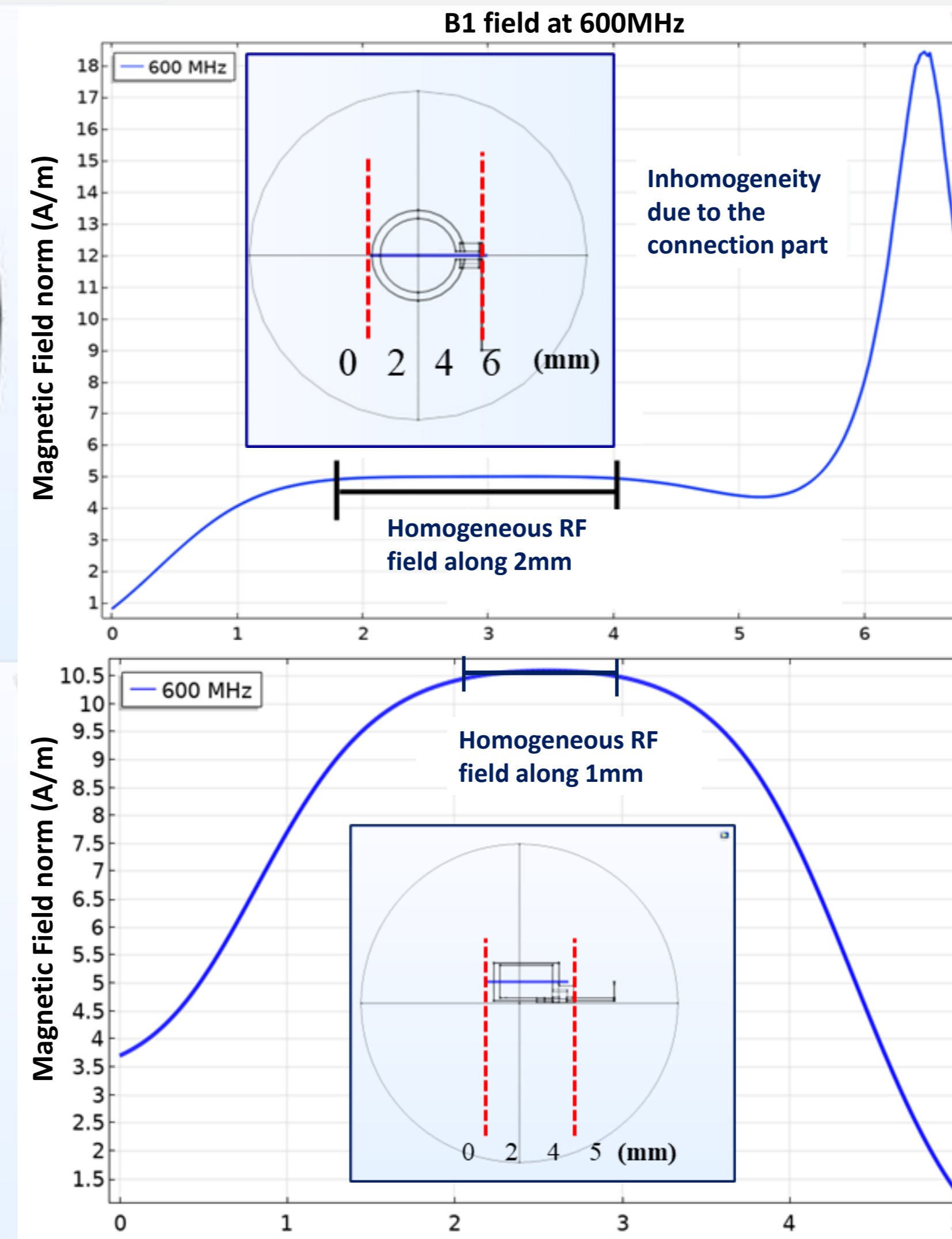
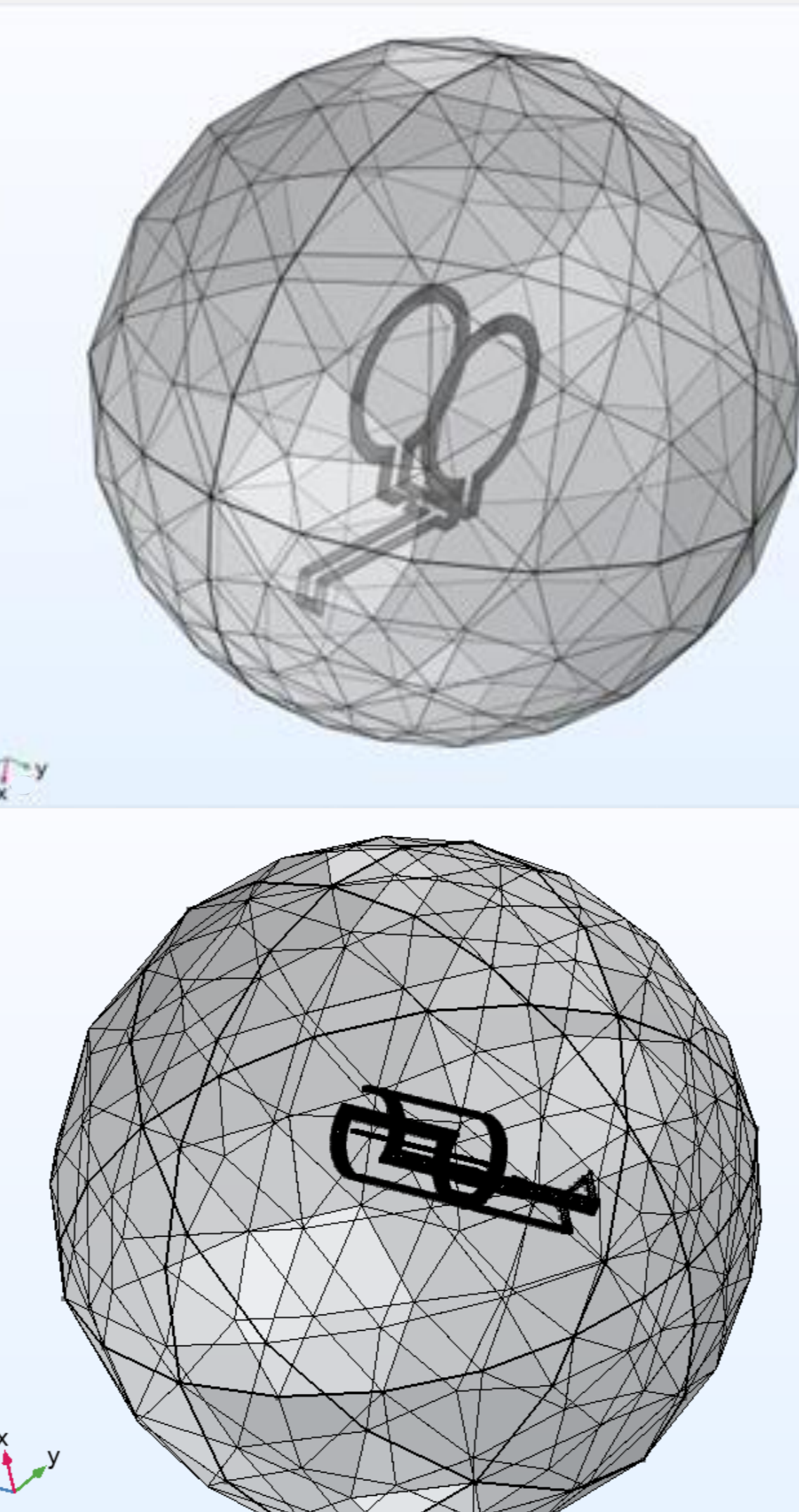


Chosen geometry for application 3.b



### • RF simulations on COMSOL Multiphysics®

Physics : Radiofrequency and electromagnetic wave  
Calculation: Mesh generated by the finite element method

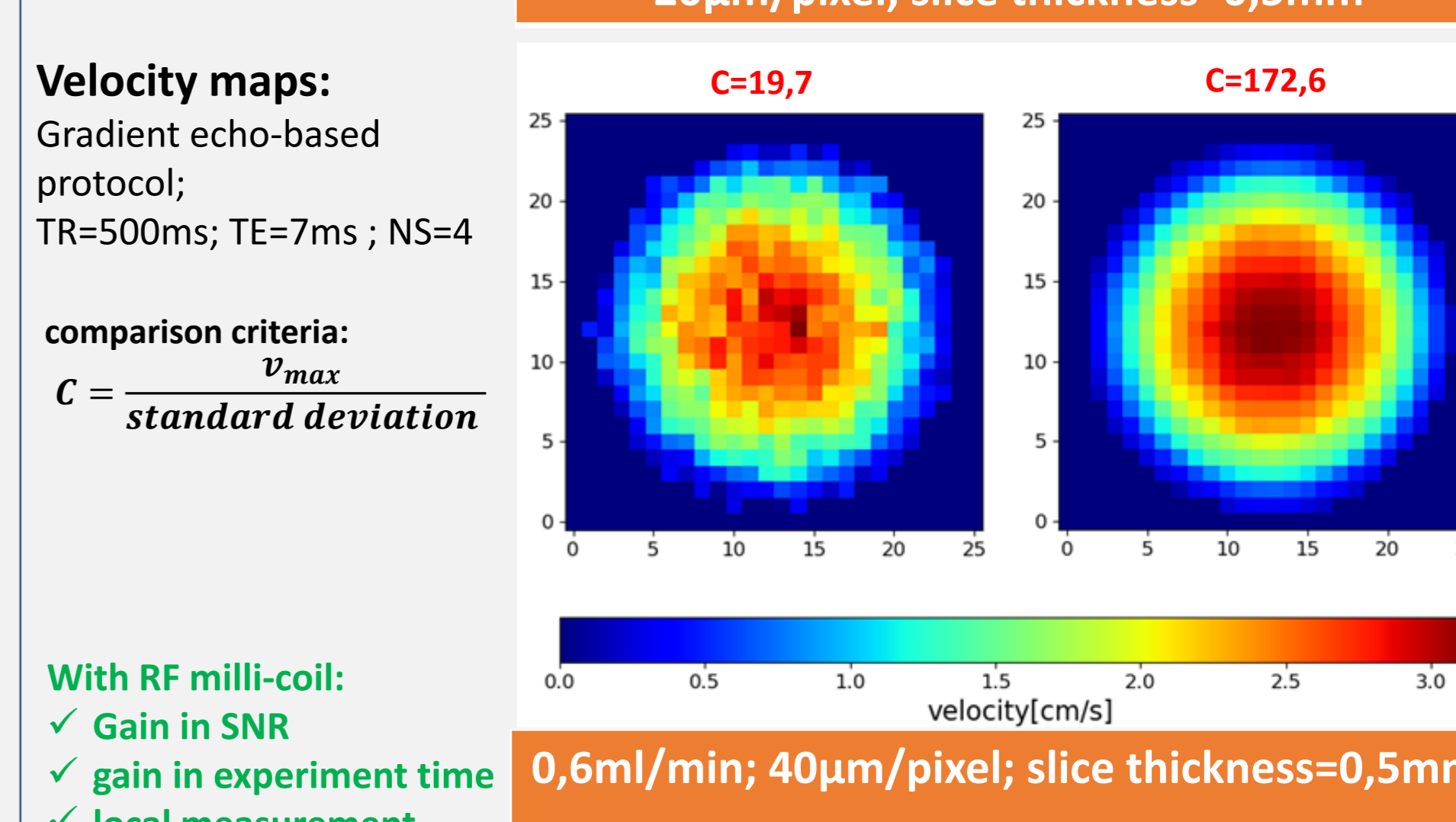
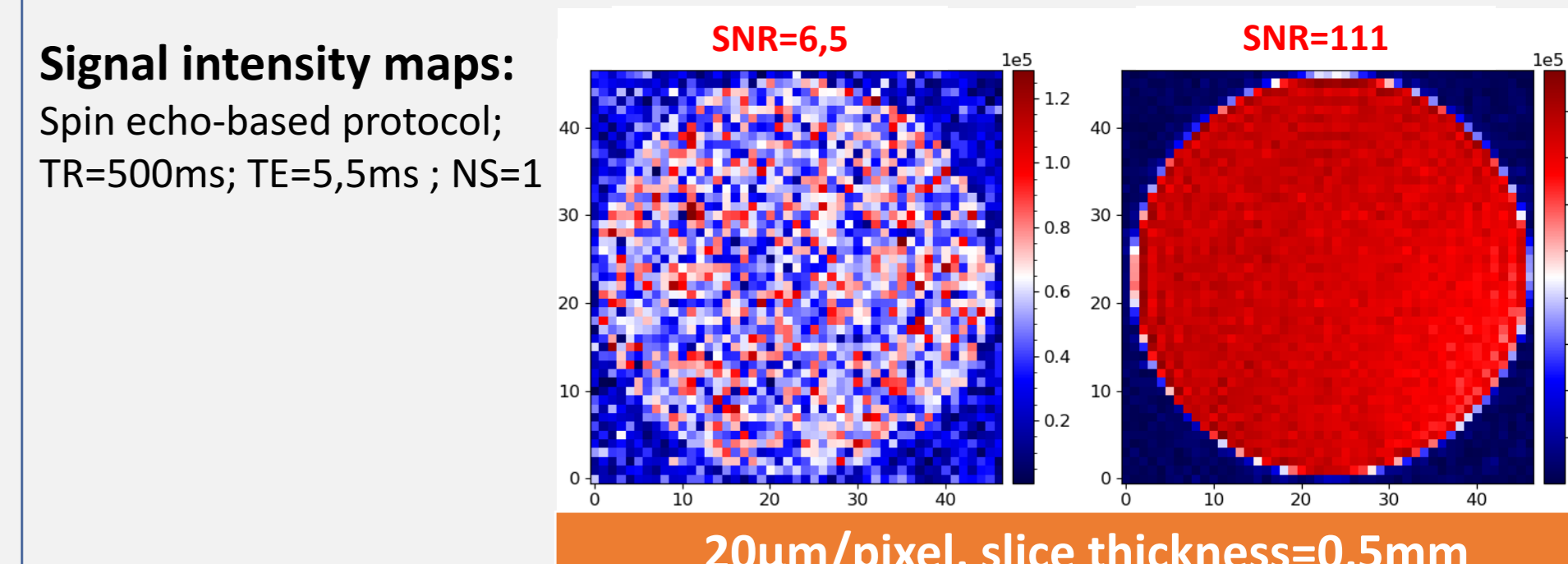


### • Qualification

Comparison measurements between a commercial probe (Bruker MicWB40 1H, 25mm ID) and the fabricated milli-coil



Sample: Capillary 0,9mm ID + CuSO<sub>4</sub> solution (T<sub>1</sub> = 100ms)



comparison criteria:  
 $C = \frac{v_{max}}{standard\ deviation}$

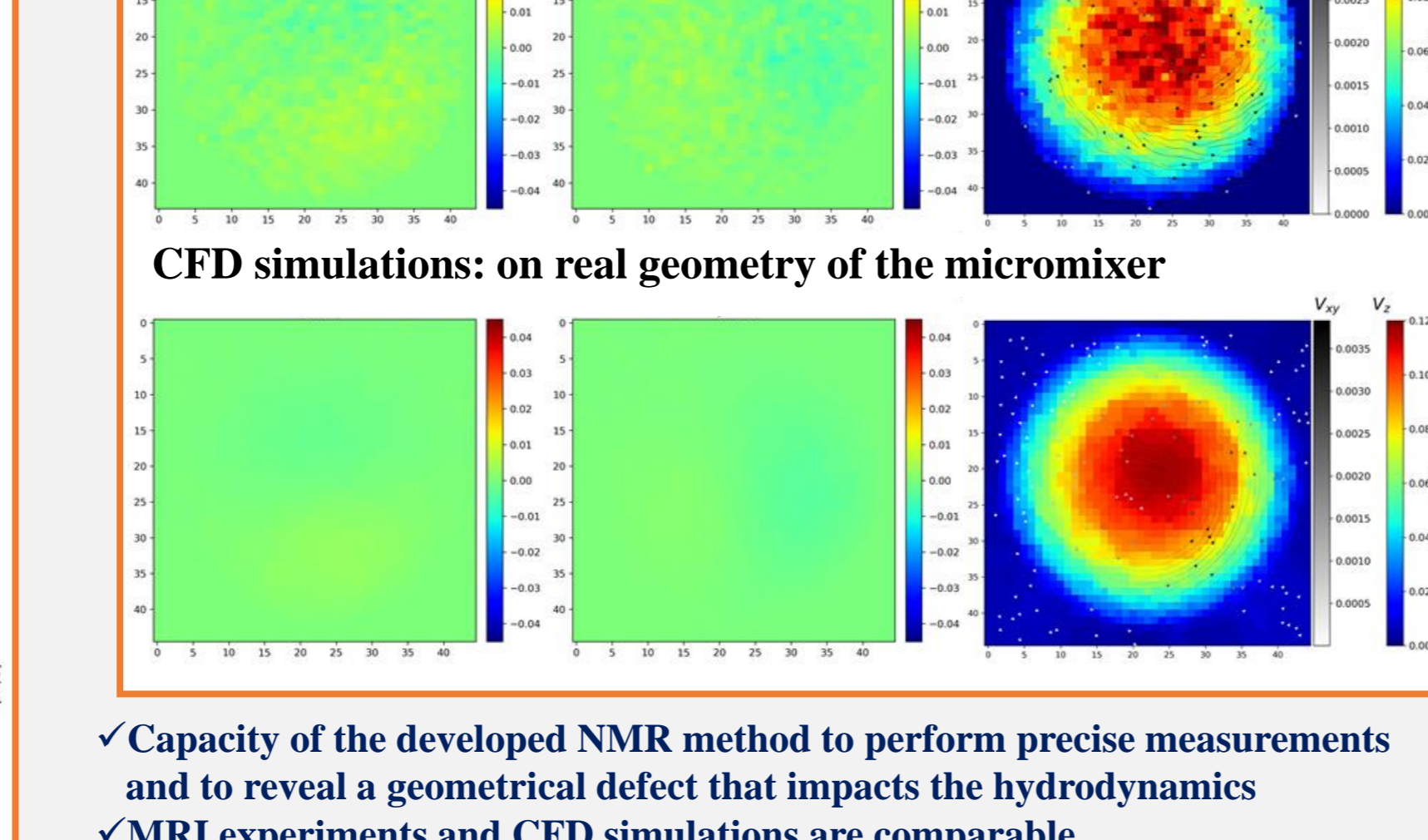
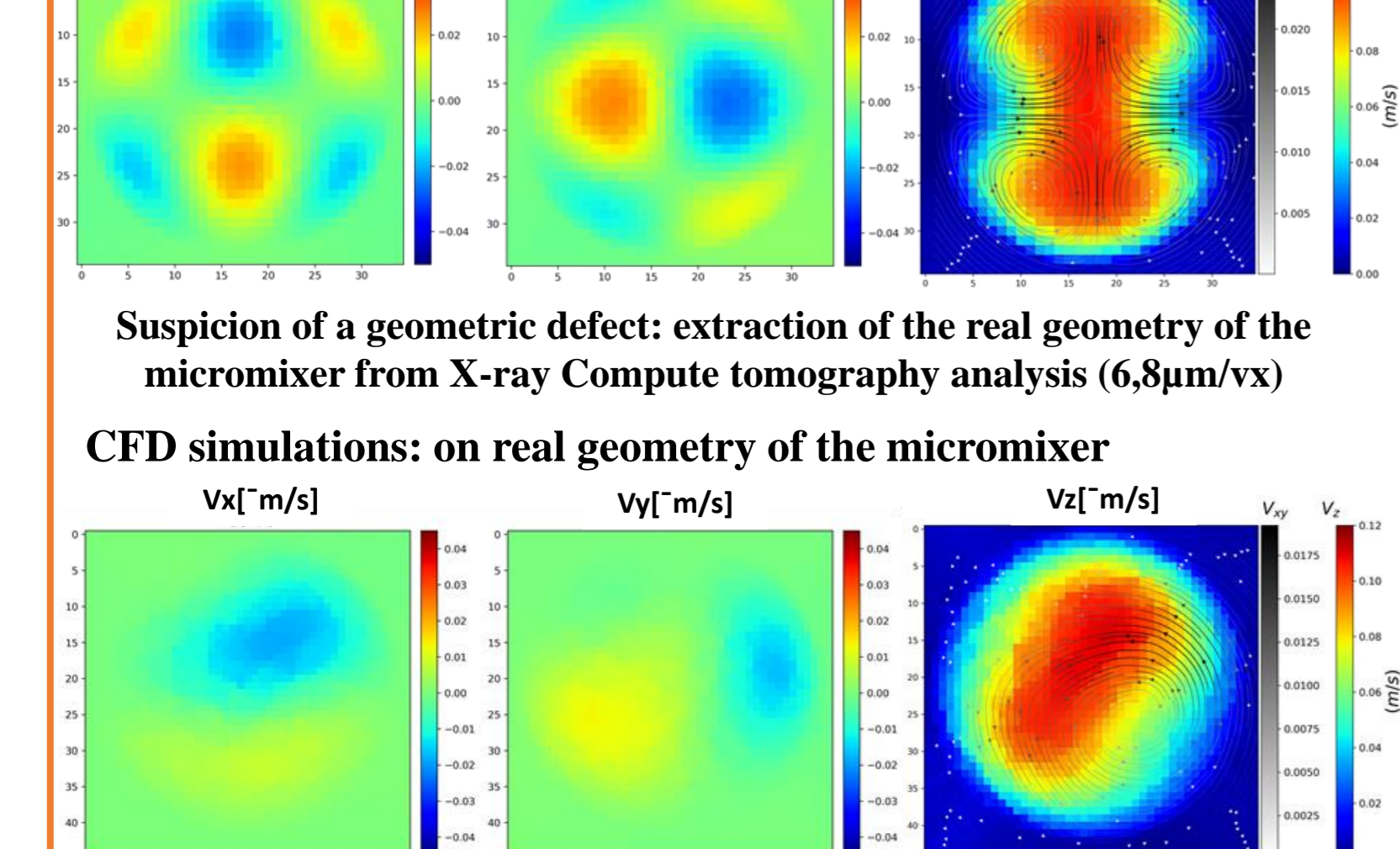
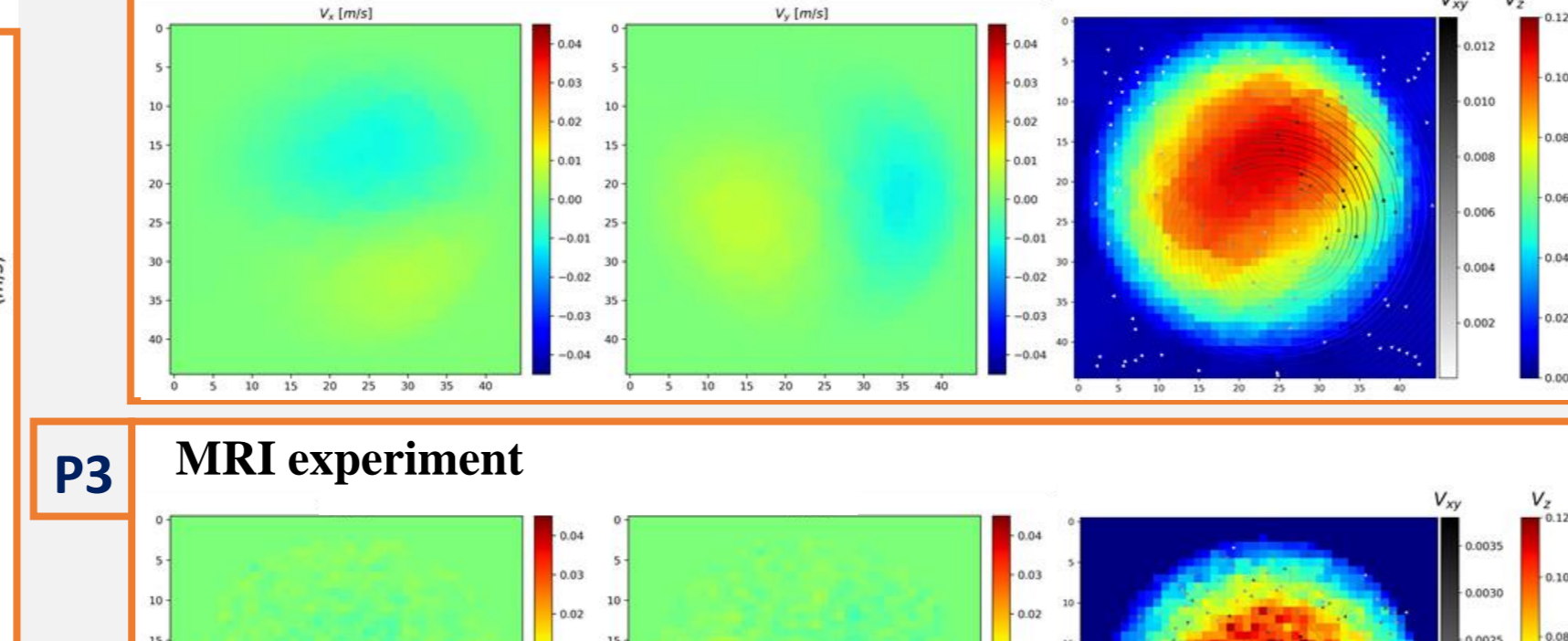
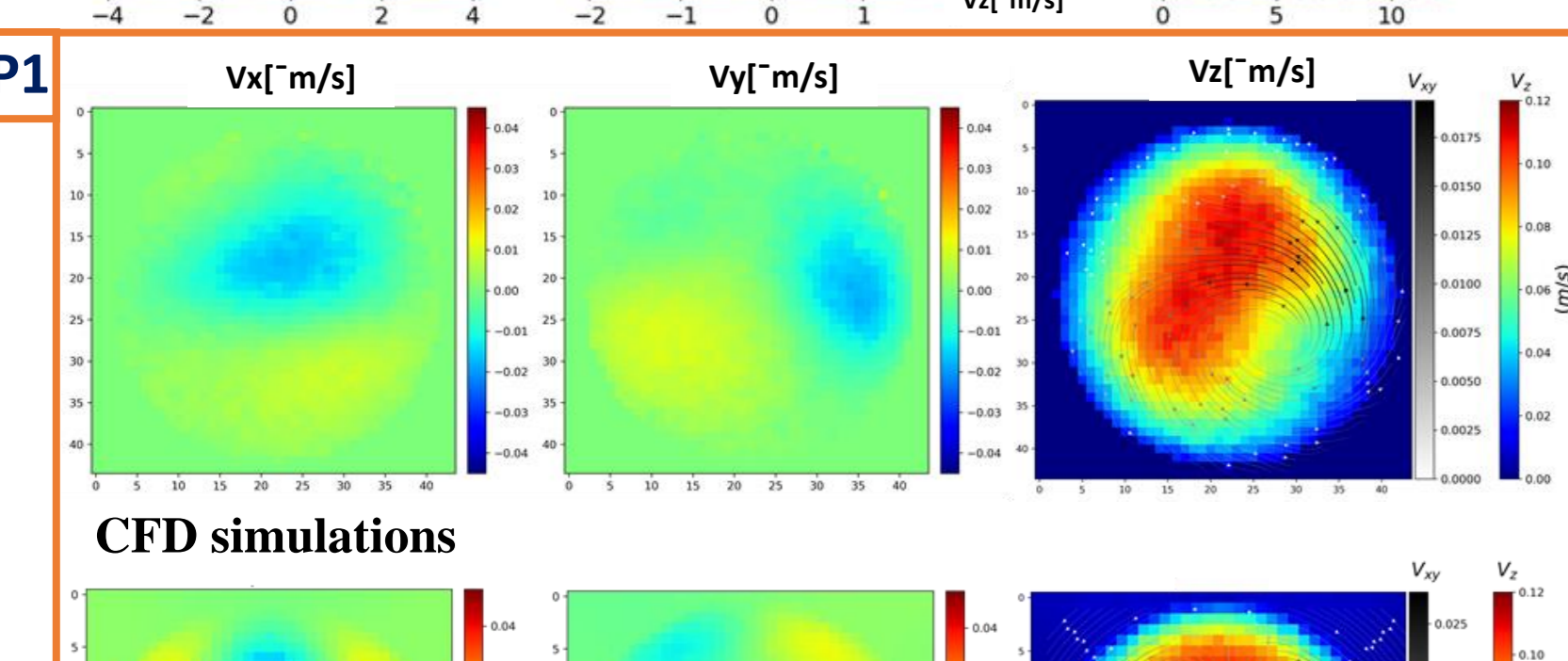
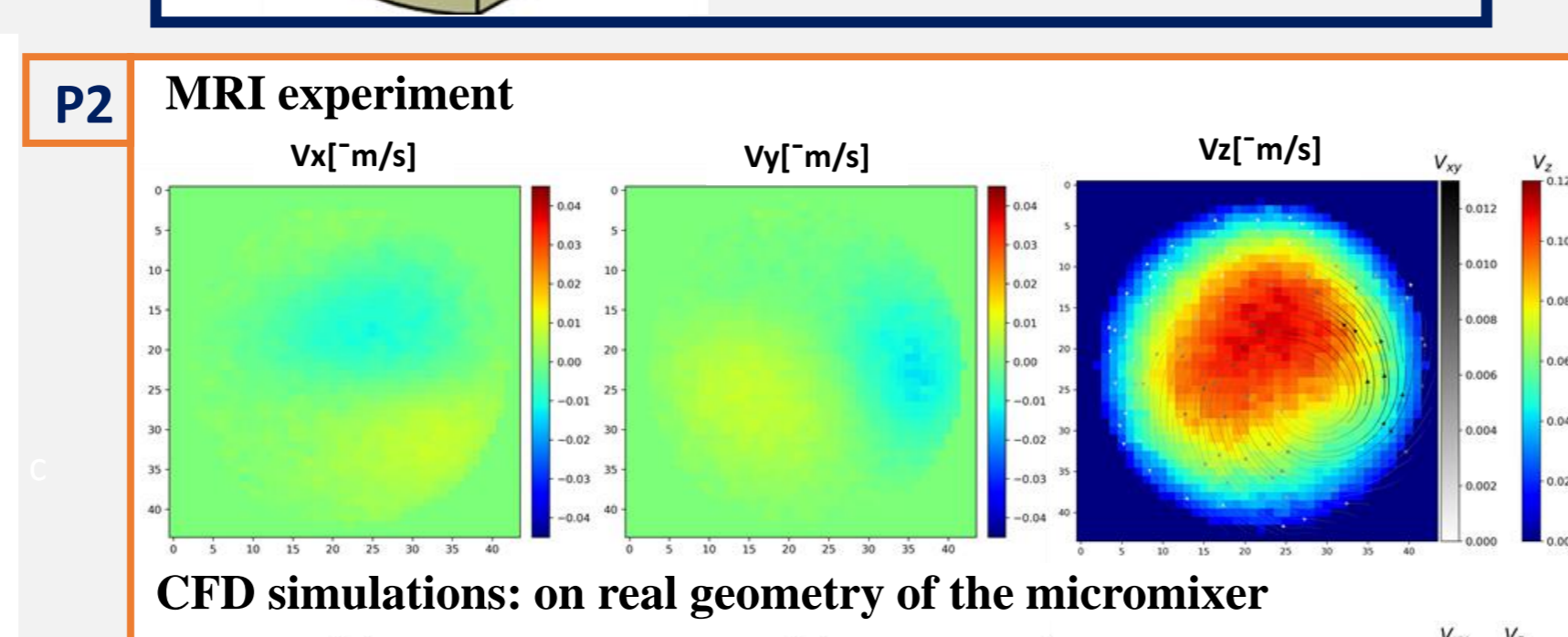
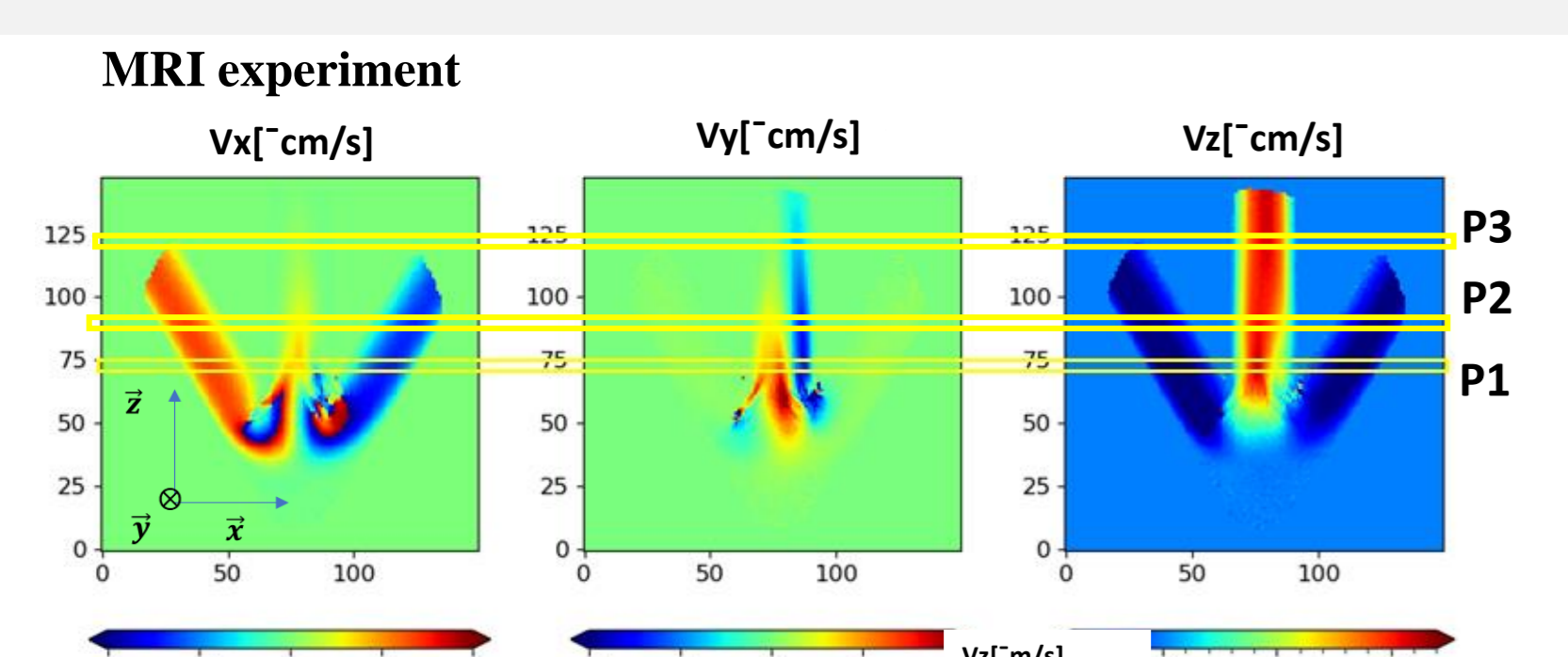
With RF milli-coil:  
✓ Gain in SNR  
✓ gain in experiment time  
✓ local measurement

## 3. Results

### a. MRI study of the hydrodynamics in micro-mixers

- Characterization of hydrodynamics instabilities in a W-shaped micromixer (Re per entry ~ 100 ) by a coupled approach :
  - ✓ MRI measurements
  - ✓ Numerical simulations<sup>7</sup> (CFD- Ansys Fluent)
- Comparison of the two approaches
- Validation of the numerical simulations

Fluid/entry	CuSO <sub>4</sub> solution (T <sub>1</sub> = 100ms)
Re/entry	60 to 170
Slice thickness	0,2mm
Resolution	24μm/pixel
MRI protocol	Gradient echo;; TE=5,5ms



✓ Capacity of the developed NMR method to perform precise measurements and to reveal a geometrical defect that impacts the hydrodynamics  
✓ MRI experiments and CFD simulations are comparable

### b. MRI visualization of a biofilm development in a capillary

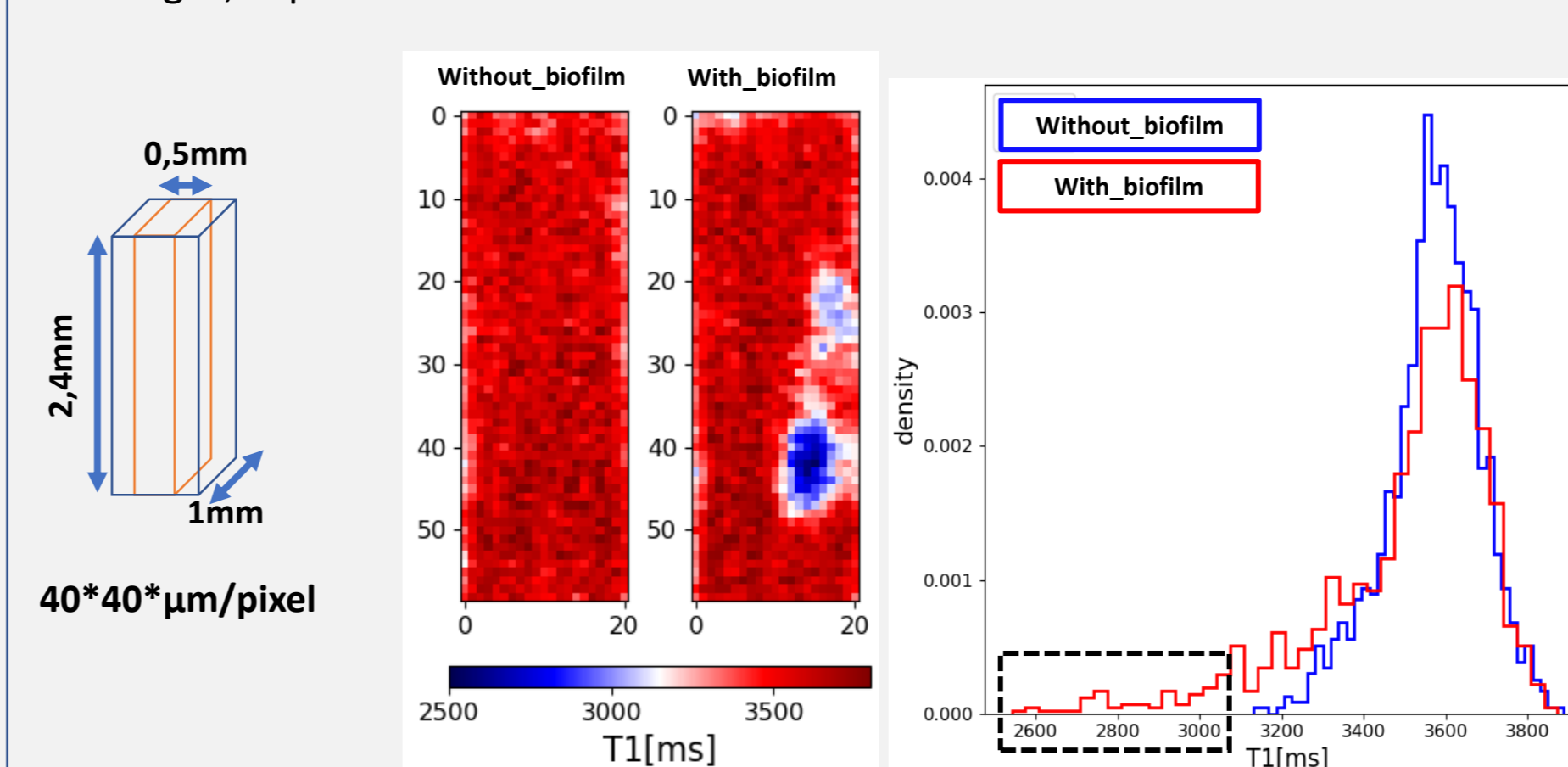
- perform a biofilm growth experiment in a capillary of submillimeter dimensions (pore model)
- MRI visualization of a biofilm and its impact on the hydrodynamics<sup>8,9</sup>

Protocol for biofilm culture:

- bacteria: shewanella oneidensis (soil model)
- adhesion stage: colonize the capillary with the bacterial culture during 4h at ambient temperature (flow 1.2ml/min, ; in closed-loop)
- biofilm culture stage: continuous feeding with nutrient solution (flow rate 1.2ml/min)

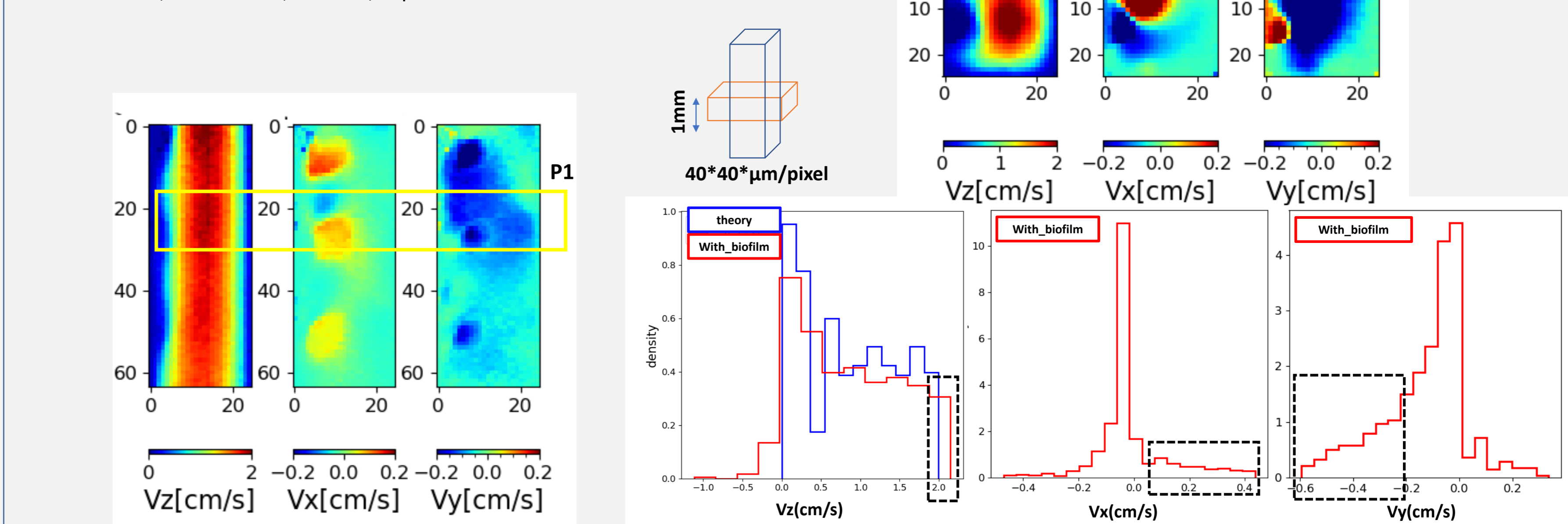
#### Longitudinal relaxation measurements

Spin echo; protocol RARE VTR; TE=10ms; 10 TR [100-10000ms]; RARE Factor=2; 2 averages; experiment time 40min



#### Velocity measurements

Gradient echo; TR=1000ms; TE=7ms; experiment time 6min



✓ Test of a biofilm growth experiment at the pore scale  
✓ Visualize by MRI a biofilm development and its impact on hydrodynamics

## 4. Conclusions

- We developed a MRI instrumentation and implemented milli-fluidic devices with simple, fast prototypes and low-cost materials
- We improved the signal to noise ratio by the development of RF milli-coils
- These methods perform precise measurements of milli-fluidic phenomena which allows the inspection of complex systems

(1) Chen, L.; Yang, C.; Xiao, Y.; Yan, X.; Hu, L.; Eggersdorfer, M.; Chen, D.; Weitz, D. A.; Ye, F. Millifluidics, Microfluidics, and Nanofluidics: Manipulating Fluids at Varying Length Scales. *Materials Today Nano* 2021, 16, 100136.  
(2) Miao, A.; Anderson, R. C., *Microsystem Technology in Chemistry and Life Science*; Springer: Berlin Heidelberg, 1998.  
(3) McDonnell, E. E.; Han, S.; Hilly, C.; Pierce, K. L.; Pines, A. NMR Analysis on Microfluidic Devices by Remote Detection. *Anal. Chem.* 2005, 77, 8109-8114.  
(4) Montinaro, E.; Grisi, M.; Letizia, M. C.; Pethö, L.; Gijjs, M. A. M.; Guidetti, R.; Michler, J.; Brugger, J.; Boero, G. 3D Printed Microchannels for Sub-NL NMR Spectroscopy. *PLoS ONE* 2018, 13 (5), e0192780.  
(5) Massin, C.; Vincent, F.; Homay, A.; Ehrmann, K.; Boero, G.; Besse, P.-A.; Daridon, A.; Verspoorte, E.; de Rooij, N. F.; Popovic, R. S. Planar Microcoil-Based Microfluidic NMR Probes. *Journal of Magnetic Resonance* 2003, 164 (2), 242-255.  
(6) Webb, A. G. Radiofrequency Microcoils for Magnetic Resonance Imaging and Spectroscopy. *Journal of Magnetic Resonance* 2013, 229, 55-66.  
(7) Engler, M.; Kockmann, N.; Kiefer, T.; Waiss, P. Numerical and Experimental Investigations on Liquid Mixing in Static Micromixers. *Chemical Engineering Journal* 2004, 101 (1-3), 315-322.  
(8) Herrling, M. P.; Lackner, S.; Nirschl, H.; Horn, H.; Guthausen, G. Recent NMR/MRI Studies of Biofilm Structures and Dynamics. In *Annual Reports on NMR Spectroscopy*; Elsevier, 2019; Vol. 97, pp 163-213.  
(9) Seymour, J. D.; Gage, J. P.; Codd, S. L.; Gerlach, R. Anomalous Fluid Transport in Porous Media Induced by Biofilm Growth. *Phys. Rev. Lett.* 2004, 93 (19), 198103.