HANDBOOK OF
FLUID – POROUS MEDIA
INTERACTIONS

InterPore BeNeLux chapter

PREFACE

The *frontiers of knowledge* are always on the move. What is today a discovery, tomorrow it will be common knowledge at academia level or even for industrial applications. The processes of advancing the frontiers, as well as of using the new discoveries in a beneficial manner for society, take place in stages. The first stage is to prepare an inventory of what is known in a well-defined subject area (e.g. fluids-porous media interactions), which may form the grounds for new ideas to germinate. This is the aim of this digital “Handbook of Fluid – Porous Media Interactions”.

As an important branch of the InterPore Society, *the BeNeLux Chapter of InterPore* acts as a platform for scientists, engineers, and managers involved in porous media systems. Our aim is to promote advancement and dissemination of knowledge for the understanding, modeling and description of natural and industrial porous media systems and associated processes.

The “Handbook of Fluid – Porous Media Interactions” gives an overview of research facilities that exist at the research groups from academia or industry in the BeNeLux. More than this, the Handbook is a great facilitator for new research collaborations. We consider it to be a living document, which periodically will be updated with new information and with new research groups. The ownership and the copyright of all material provided here belong to the information provider; also the responsibility for the scientific content resides with them. The InterPore chapter is a facilitator to keep this Handbook project going.

The Steering Committee of the BeNeLux InterPore Chapter takes the opportunity to thank the present and the future contributors.

Nicolae Tomozeiu,
25 November 2019

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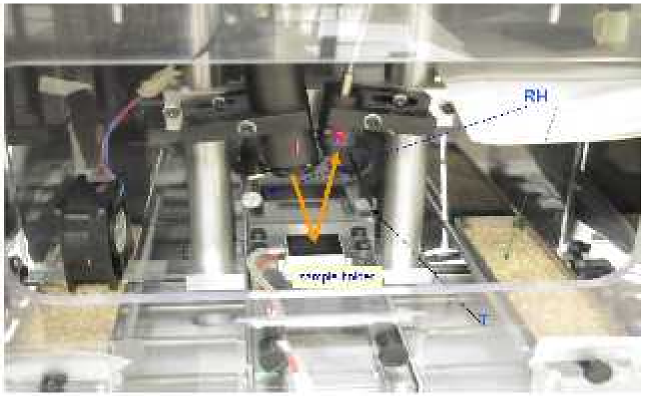
VIS Reflectometry – for thin liquid films drying

What: Characterize the physical processes of a thin film of liquid during drying

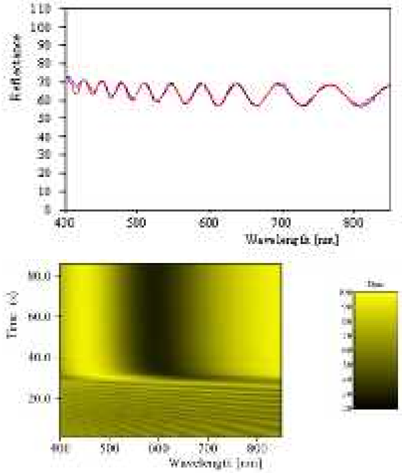
Example: liquid evaporation, latex film formation from water-latex mixtures

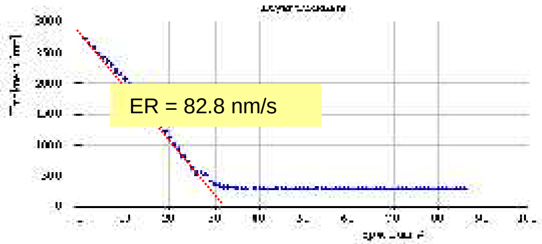
How: VIS reflection spectra: experimental measurements + theory in data analyzing

Setup



Results and Analysis





A thin liquid film of water + latex particles + surfactant is formed on a c-Si sample holder. The relative humidity and temperature are controlled. Measuring the spectra with a periodicity in time, the sample evolution is revealed by 3-D like plots. Via spectra simulation, the evaporation rate is calculated.

Limits of the method: the substrate must reflect the VIS light, no rough substrates, the scattering is neglected

IR Spectroscopy - for composition analysis

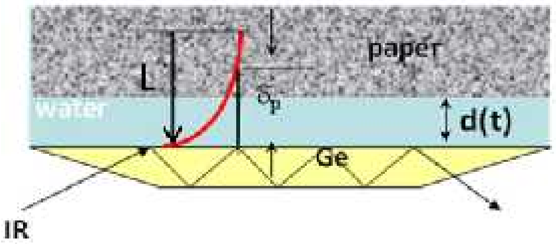
What: Characterize static and/or dynamics of material composition during various physical-chemical processes

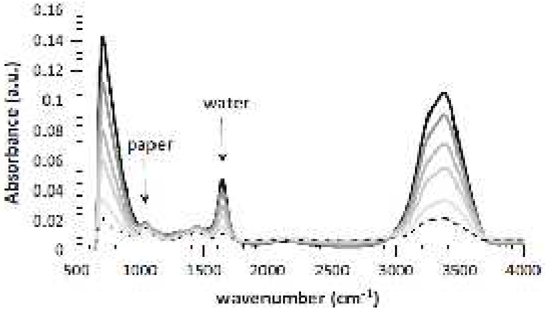
Example: material characterization, process evaluation: evaporation, liquid imbibition

How: IR absorption and/or ATR spectroscopy

Setup

Results and Analysis





A layer of complex liquid is placed on the ATR crystal; if the liquid top-surface is free, the measured IR spectra will provide useful data regarding the evaporation process. If the liquid layer is covered by a porous material and the evaporation is canceled, the IR spectra reveal the liquid penetration into porous media process. Measuring the spectra with a periodicity in time, the dynamics of the processes is shown.

Limits of the method: Although the method allows studying the penetration of each component of the complex liquid, grace of well-defined molecular fingerprints, it is an indirect method that probes a limited volume of the investigated sample

Automatic Scanning Absorptometer (ASA)

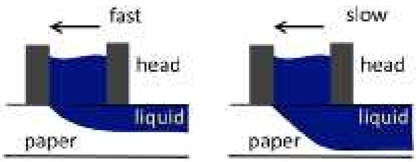
What: quantitatively monitoring the liquid penetration into porous material

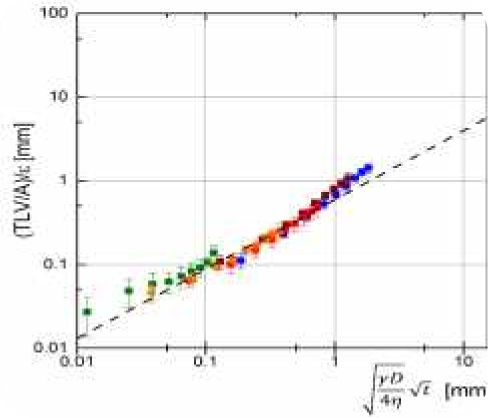
Example: liquid permeability of porous material, absorption rate;

How: Based on Bristow method, ASA accurately determines the liquid penetration into porous media on timescales of milliseconds to several seconds

Setup

Results and Analysis





The porous sample is placed on a turntable and via a nozzle from a scanning head the liquid is supplied and penetrates into the sample due to capillary suction. A sensor measures the absorbed liquid volume on a glass capillary.

Limits of the method: the limited size of the liquid reservoir influences the maximum contact time for measurements

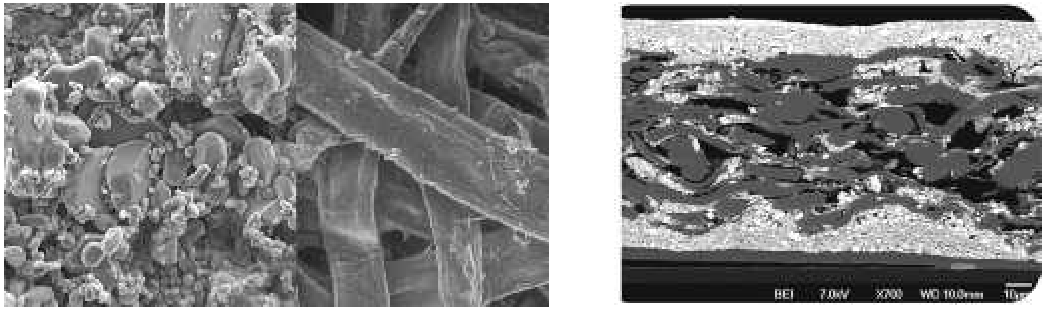
SEM Spectroscopy

What: external morphology (texture), chemical composition, structural characteristics on surface and cross-section of the sample

How: Secondary electrons (SE) and backscattered electrons (BSE) are used for imaging samples: SE reveal morphology and topography, while BSE shows contrasts in composition in multiphase samples.

Setup

Results and Analysis



Surface morphology of a coated paper and a plain paper revealed with high resolution SEM. Using special preparation of the sample (e.g. ion beam polishing) the distribution of various structural entities in the sample cross-section can be studied.

Limits of the method: Samples must be solid and small dimension; "wet" samples can be studied in "low vacuum" SEM setup. An electrically conductive coating must be applied to insulating samples.

Rheology

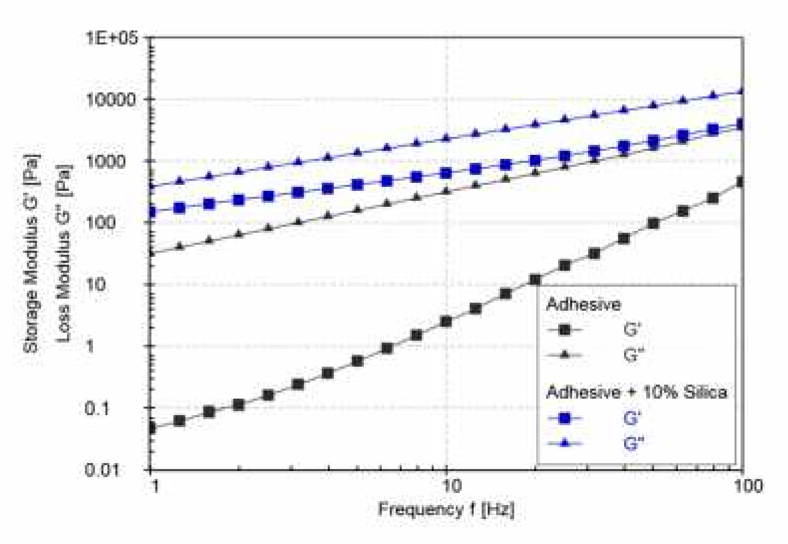
What: deformation and flow of materials that have a complex microstructure, both solids and liquids;

Example: liquid viscosity, particles interactions during phase transformation

How: Rheometers impose a specific stress field or deformation to the sample, and monitor the resultant deformation or stress. They can be run in steady flow or oscillatory flow (shear or extension).

Setup

Results and Analysis



Rheological properties are strongly temperature and frequency dependent.

An oscillatory measurement is used to analyze the effect of the addition of silica used to modify the flow properties of an uncured adhesive.

Temperature = 25ºC

Limits of the method: sample volume of about 1 ml is required. For solid materials a different setup is used.

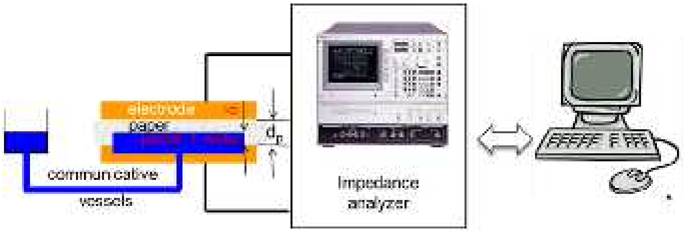
Electrical Impedance Spectroscopy (EIS)

What: material characterization, processes dynamics;

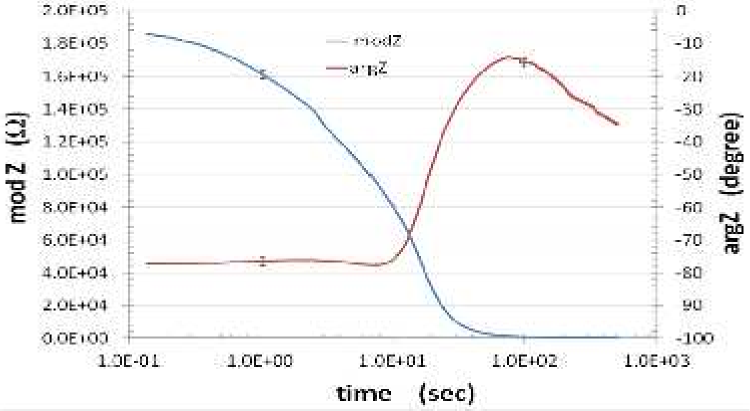
Example: dielectric constant, liquid evaporation, liquid imbibition into porous materials

How: a.c. measurements (frequency varies between 1 kHz and 10 MHz) provide the modulus and the argument of the electrical impedance. These physical amounts are sensitive to the sample composition

Setup



Results and Analysis



The variation in time of the modulus and argument measured on a porous paper (here displayed for f= 10 kHz) reveals the water imbibition process due to capillary suction.

$$\in(\omega) = \in_1(\omega) - j\in_2(\omega) = \frac{}{\cdot\mu\cdot \quad \cdot \; (\mu)}$$

Limits of the method: the dielectric constant of the liquid must be different than the dielectric constant of the porous material

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Physics of Fluids

msm

University of Twente is an entrepreneurial research university, dealing with theoretical, numerical and experimental studies. It comprises several research groups and departments, working on porous-fluid interactions.

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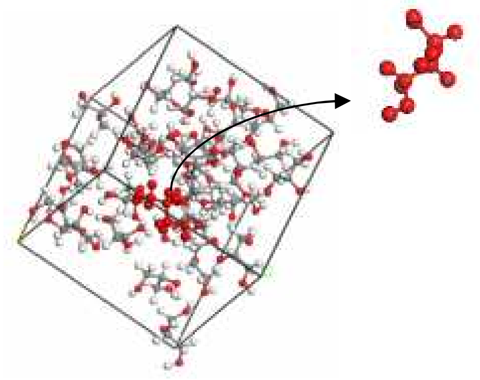
Ink formulation using Hansen solubility parameter

What: Prediction of the wettability of ink as well as the miscibility between the ink components

Example: Ink components miscibility, Ink wettability, pigments solubility.

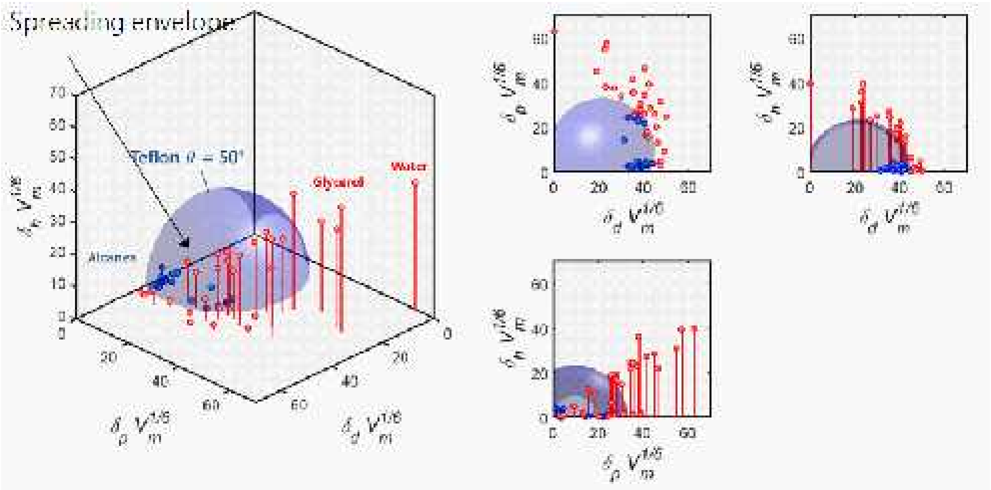
How: Since the solubility parameter contains all the energies holding the liquid together, it represents an important descriptor for droplet wettability and the interactions between the ink components.

Modeling programs and Tools



MD simulations using LAMMPS and group contribution methods, and post processing using Matlab.

Results and Analysis



Components inside the spreading envelope (in blue) will tend to spread on the substrate.

Limits of the method: Group contribution methods are fast, but not as accurate as MD. MD is slower but more accurate and universal.

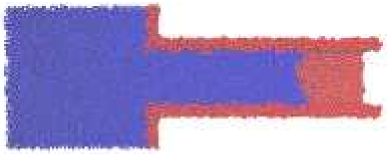
Mesoscale modeling of ink-Imbibition in paper

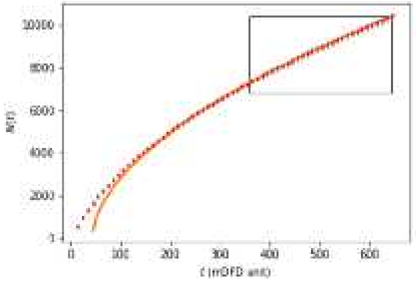
What: Perform mesoscopic simulations of the ink penetration process in paper

Example: Ink flow, liquid imbibition.

How: Using a particle based simulation technique called many-body dissipative particle dynamics (mDPD)

Modeling programs and



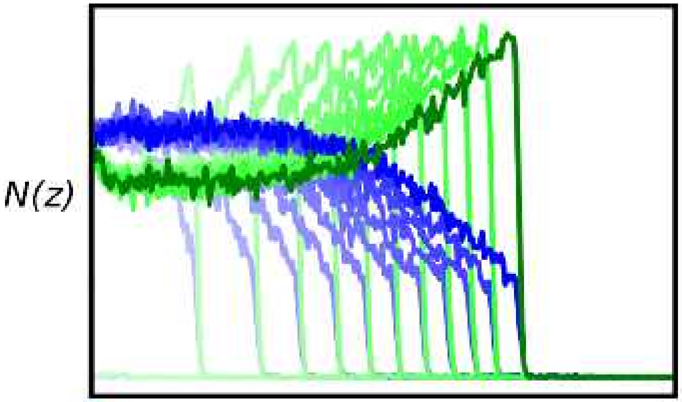


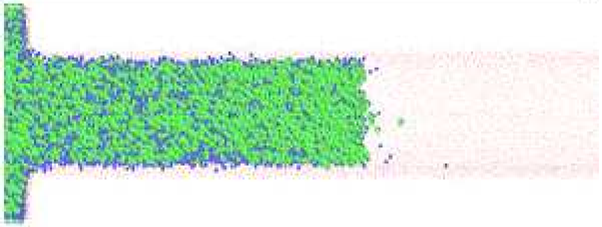
mDPD was used for the simulation. The setup consists of calibrating the surface tension, contact angle and the viscosity of a model fluid. Following this, the square of the imbibition depth of a model fluid inside a nanochannel is found to increase linearly with time, as per the Lucas-Washburn law.

Results and Analysis

Ink is a complex fluid containing multiple components. We first investigate the imbibition of binary miscible fluid mixtures (blue + green) in a nano-channel. Blue component has a greater wettability, but identical viscosity as the green component.

The complexity of the solid will be modeled in the future.





Limits of the method: Even though the mDPD method is well suited for pore-scale simulations, the method cannot capture the entire pore-size distribution as found in paper.

Immiscible displacement in porous media microfluidic chip

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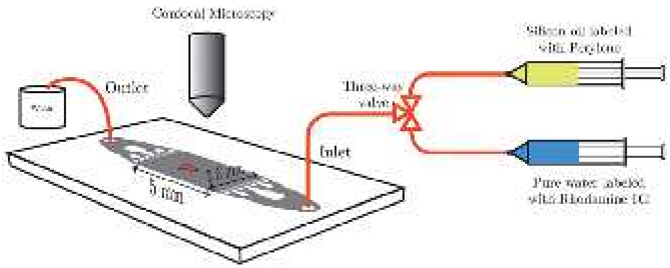
What: Observation of liquid-liquid displacement in a microfluidic chip mimicking porous media

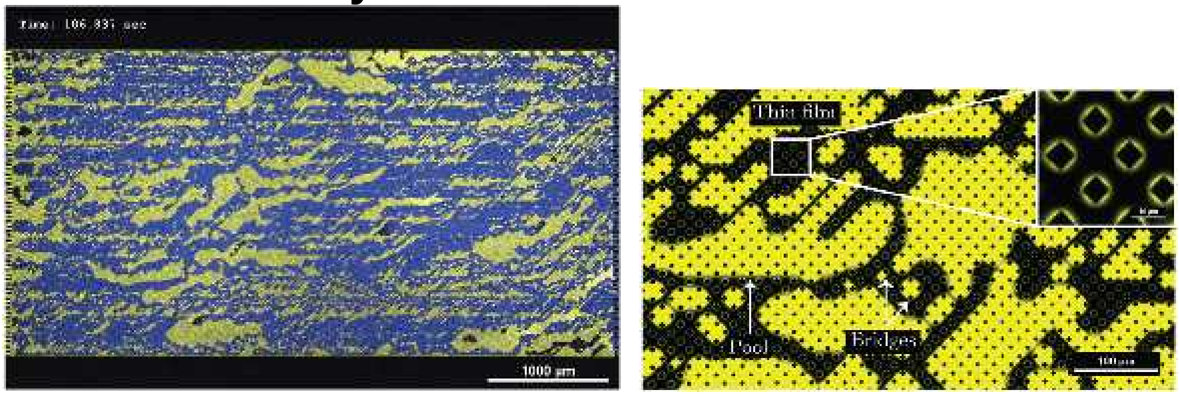
Example: Enhanced oil recovery, immiscible displacement in porous media such as porous membrane

How: A microfluidic chip consisting regular arrays of square pillars were fabricated from silicon wafer and hydrophobized. Then the chip was filled with silicone oil and water was pushed through at different flow rates. The observation was done using laser scanning confocal microscope [1].

Setup



Results and Analysis



(a) Image after water transport through the liquid infused chip at $Q = 0.2$ «l s^{-1} (Ca = 1.23 ò 10^{-4}) (yellow color is the oil phase and blue color is the water phase). (b) Image showing different configurations of the residual wetting fluid [1].

Limits of the method: The chip was not a resemblance of a real porous media and designs with random arrays of pillars are needed.

[1] Bazyar, H.; Lv, P.; Wood, J. A.; Porada, S.; Lohse, D.; Lammertink, R. G. H. Liquid-liquid displacement in slippery liquid-infused membranes (SLIMs). *Soft Matter* **2018**, *14*, 1780–1788.

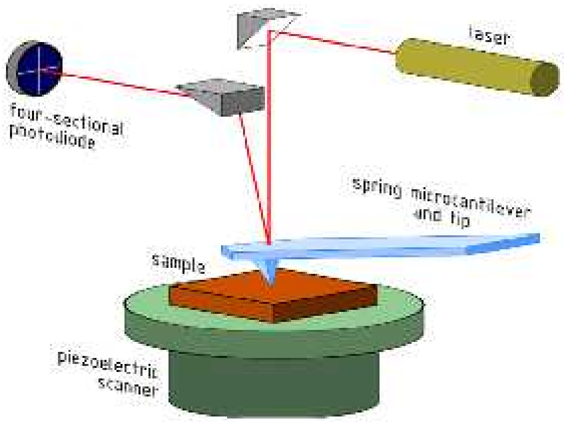
Atomic force microscopy

What: Topography of solid surfaces, mechanical properties on a micro/nano-scale;

Example: Silicon surface topography, molecules adsorption on surfaces.

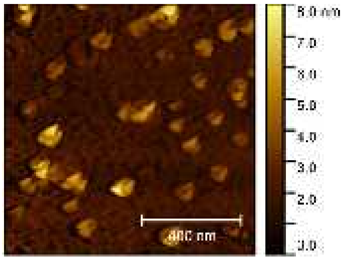
How: Very sharp tip fabricated on a cantilever is interacting with a surface in different modes (tapping, contact, force-distance etc.) providing information about its topography and properties

Setup



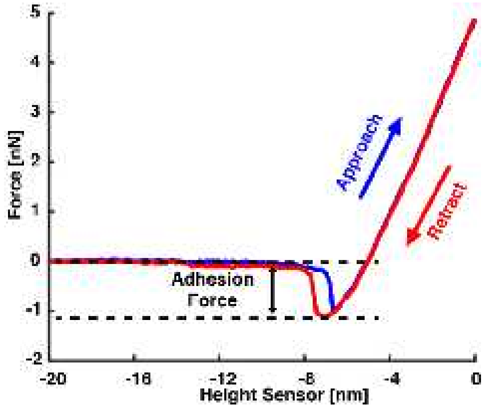
Results and Analysis

Apart of topographical (below) images of the surface AFM can provide information about mechanical and electronic properties of the surface. Data about mechanical properties can be extracted from the force-distance curves (right).



Topographical image of clean glass surface. Crystalline domains are clearly visible.

Typical force–distance curves obtained with the AFM



Limits of the method: Height changes on the sample cannot be too large. Only local information. Measurements are strongly probe (tip) dependent.

Ellipsometry

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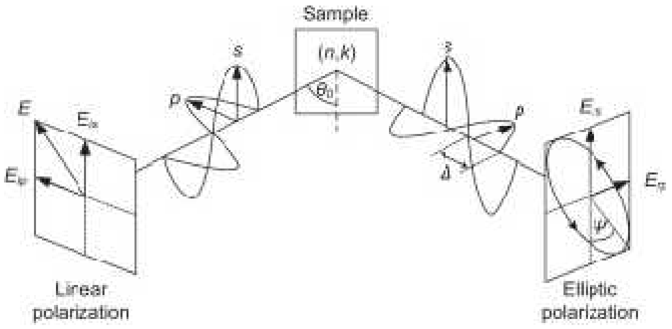
What: Measure thickness of thin films and layers;

Example: Adsorption of molecules on surfaces, layered structures.

How: Polarized light is reflected from the sample at different angles for different wavelengths. Change of polarization properties upon reflection is measured.

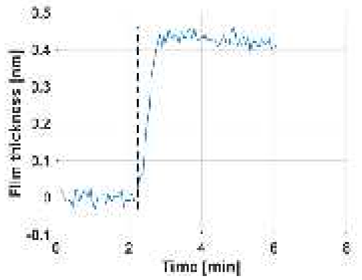
Setup



Working principle of ellipsometry. Delta and Psi parameters are measured.

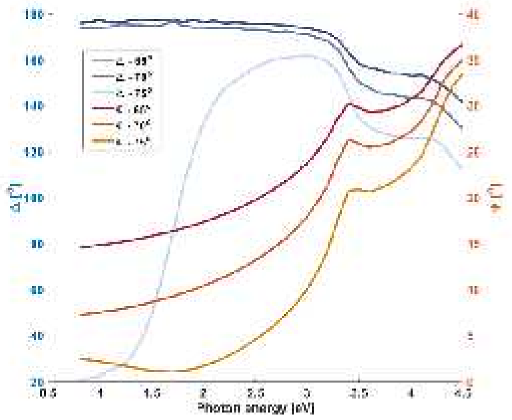
Results and Analysis

Ellipsometry can very precisely (up to 0.1 nm) measure the thickness of thin films and layers. The data analysis is based on a model created based on an optical properties of the materials measured.



Adsorption of molecules on a silicon oxide surface visualized with ellipsometry.

Typical spectrum obtained for a layer of native SiO_2 on Si



Limits of the method: Sample has to be reflective. Data analysis is based on the model, thus the information on the materials creating the film is required.

Mid-air collisions of different surface tension droplets

What: Study the collision behavior of droplets with different surface tension;

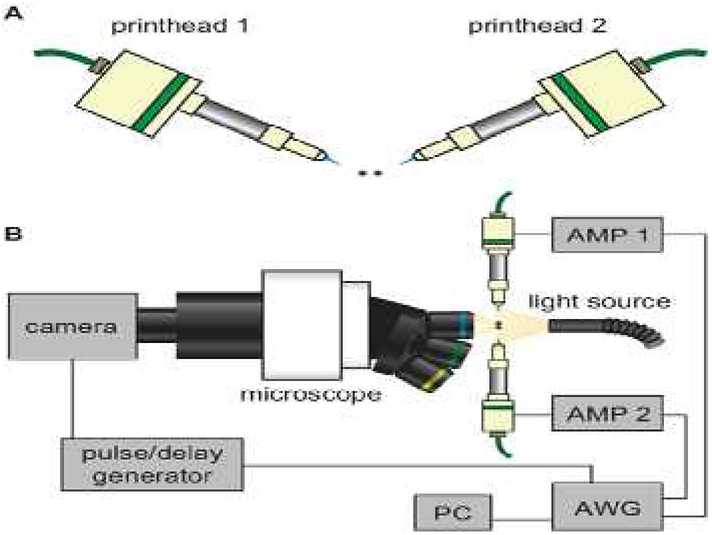
Example: Droplets, Mid-air collisions.

How: high speed imaging and image analysis of collisions.

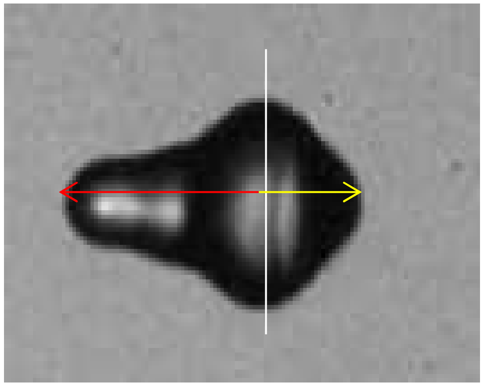
Setup

a

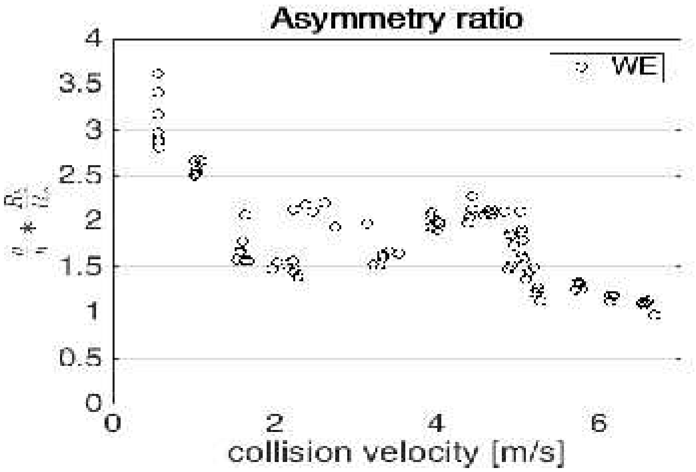
b



Results and Analysis



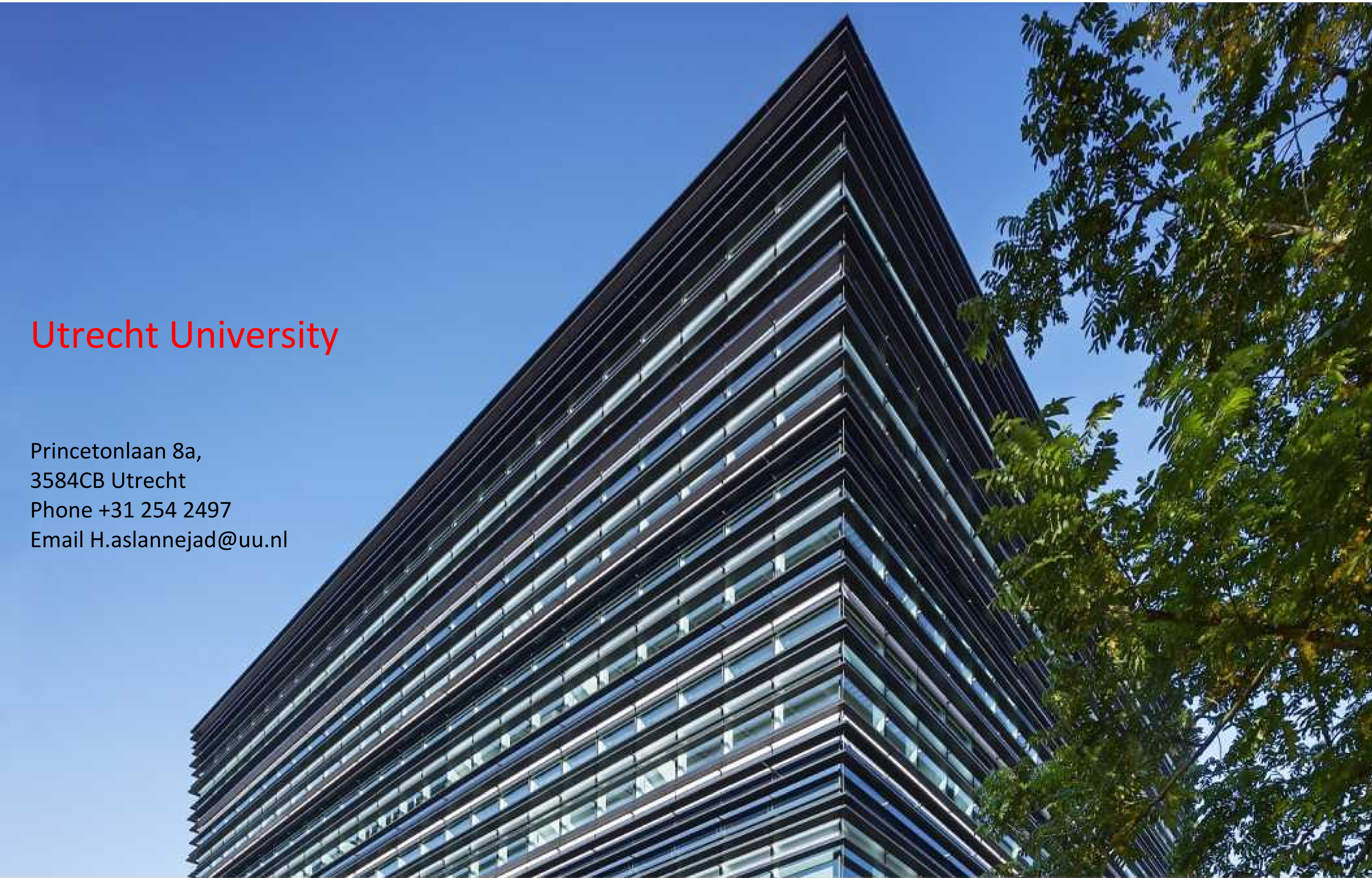
Snapshot of collision between water and ethanol droplet



Asymmetry ratio versus collision impact velocity for water-ethanol collisions

Limits of the method: Not every collision is perfectly head-on, due to droplets continuously adjusting their position.

UU MULTISCALE POROUS MEDIA LAB



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Multiscale Porous Media Lab

Research

The Multiscale Porous Media Lab is a state-of-the-art facility for performing research on complex and coupled multiphase flow and reactive transport in porous materials. This involves experimental work combined with real-time imaging techniques and several advanced numerical modelling techniques. Using this integrated experimental and computational laboratory, we are able to perform cutting-edge research in diverse fields of engineering, geosciences, and biomechanics. Current projects involve the study of ink penetration into paper, characterization of moisture absorbing products, unsaturated flow in a bed of highly swelling particles, and flow and transport in human bone, solute and colloid transport in soil and two-phase flow in porous media.

Funding and sponsors

ERC (European Research Council)

NWO (The Netherlands Organisation for Scientific Research)

FOM (The Foundation for Fundamental Research on Matter)

STW (The Netherlands Foundation for Technical Sciences)

Procter & Gamble (P&G) consumer goods corporation

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Shell Global solutions

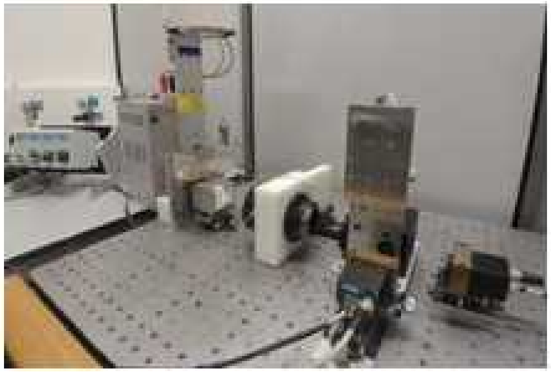
Kimberly Clark Corporation for paper-based consumer products

Facilities

Dual Energy Gamma System

Confocal Microscopy

Cleanroom is of Class 10.000



Open-air Microscope

Tank Flow Setup

Column-scale Flow Setup

Computational facilities:

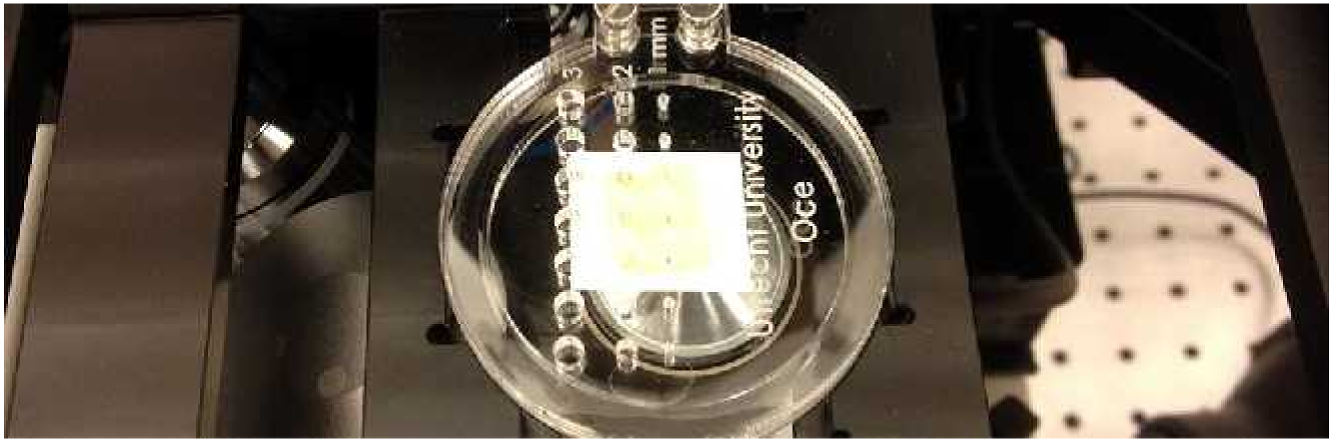
1.Cluster

Our high-performance computational cluster consists of 21 nodes, with a total of 252 CPU's. The1 cluster is used for many purposes. Ranging from use for direct numerical simulation of fluid flow in porous media to running our in-house developed software and models.

2. Workstations

We have several workstations for modeling and imaging-based applications.

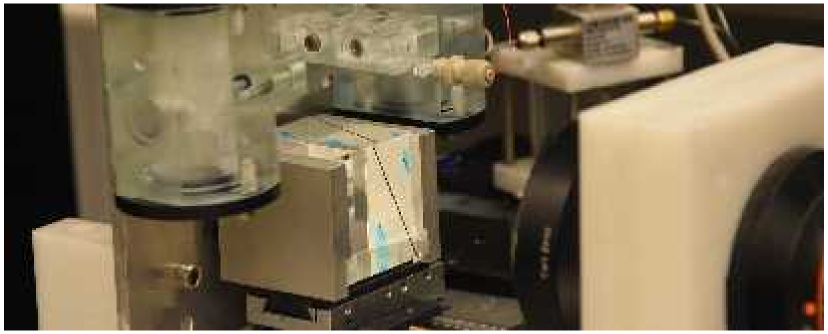
Confocal laser microscopy



Confocal scanning microscopy is an imaging modality for "optical sectioning" of a sample, that is, the imaging of thin sections at high resolution and contrast without physically dissecting the sample. Confocal microscopy offers several advantages over conventional optical microscopy, including controllable depth of field, the elimination of image degrading out-of-focus information, and the ability to collect serial optical sections from thick specimens. The key to the confocal approach is the use of spatial filtering to eliminate out-of-focus light or flare in specimens that are thicker than the plane of focus.

It is a promising technique to characterize fluid flow in porous layers, as for example in printing papers. Confocal microscopy can provide information about the pore structure in 3D and imbibition phenomena of a liquid into the pores. Basically, the microscope can be used to image an object in 3D, in case the object is semi-transparent or the combination of the porosity and thickness of the thin porous layer is in a defined range.

Open-air microscope



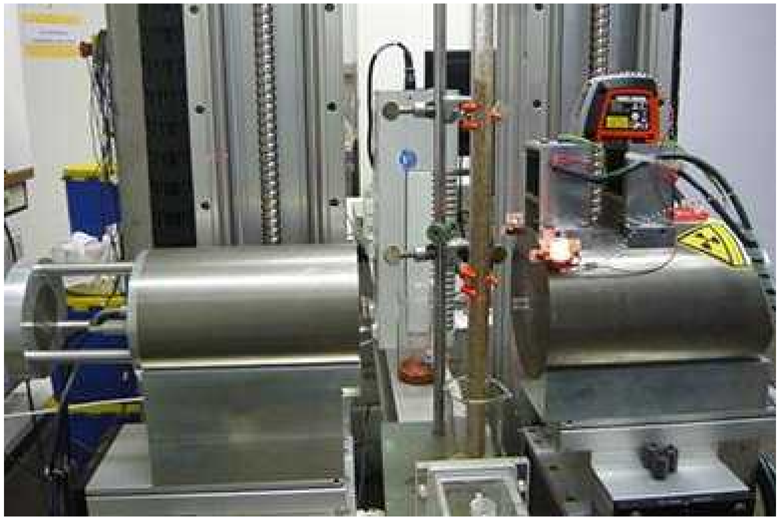
This optical set-up, designed and built inside our group, is used to visualize flow experiments in micro-models of various sizes and types. The main advantage of this set-up is that it can visualize micro-models bigger then 5x5 mm, maximum field of view, at a resolution of 2.8μm/pixel.

The set-up consists of four cameras, with resolution of 5 megapixels each, located on four sides of a box containing 3 beam splitters. The beam splitters to split the image into 2 identical ones, leading up to a total of 4 identical images, each one recorded by a camera. The incoming beam to the beam splitters has been magnified by a macro lens.

The illumination of the micromodels and the diversion of the light beam towards the objective lens depend on the type of micromodel and its refractivity. Transparent micro-models, such as PDMS, are placed between the light source and a prism that redirects the beam to the lens. On the other hand, for opaque micro-models, like Silicon-Pyrex, a beam splitter sits the between micro-model and the light source, which will divert the reflected beam to the lens.

The micro-model is placed between two pressure transducers, one in each side of the micromodel. In this way it is possible to measure, set and control the pressure between the two sides of the micromodel.

Dual-energy gamma system



Most radioactive sources can produce gamma rays, with different energies and intensities for different species. This gamma ray system has two radioactive sources, Cesium 137 and Americium 241. The opening of source is 6mm. While gamma rays emit through the source opening, the detector opposite can always measure generated spectrograms. If a sample is put between the source and the detector, the gamma rays will get attenuated when they go through the sample. Based on the decreasing of gamma rays, the length of the sample between the source and detector can be calculated. The attenuation caused by adsorption of the sample between source and detector can be described by Beer-Lambert Law. With two sources, we can easily calculate the porosities and distributions for two-phase problem in porous media.

Cleanroom

The cleanroom is of class 10.000, which is the ISO7 standard. Microfluidic micro models are made in the clean room to prevent any dust or dirt inside the model.

The room is equipped with:

- A Spin Coater
- In House made UV lithography light source
- Collection of SU-8 photoresist
    - SU-8 2025
    - SU-8 2050
    - Which can give masks with thicknesses from 22 um to 80 um and for to 40 µm to 170 µm, respectively

Focused Ion beam Scanning electron Microscopy (FIB-SEM)

For 3-dimensional imaging purposes, normally computed tomography imaging (also known as CT scan) method has been used. The imaging method is normally applied in case a resolution of few micro-meter is reasonable. However, in case the pore is so small or a resolution in the range of few nano-meter is required, FIB-SEM imaging technique can be used. The method is destructive and it basically does a series of slice&view to create stack of 2D images of the pore structure. Then, the stack of 2D images can be processed and converted to a 3D pore network. Lateral can be used in fluid flow modeling.

Modeling tools

Pore-scale modeling:
With using pore-scale modeling tools, we are able to study fluid flow into single pores of the material. Specially in case of thin layer (like paper, fuel cell, etc.), pore-scale modeling is needed because due to the lack of enough pores along the cross section of the layer, continuum scale modeling is not applicable.

a) OpenFOAM

b) Pore Network Modeling

c) DUMUX

d) Geo-dict

e) Pore unit assembly method

Continuum scale modeling:
Continuum scale modeling tools are used in case there is no need to study fluid flow interaction with single pore of the material. For instance, in field scale modeling works, the thickness of the layer is large enough to include enough REVs and therefore, averaged values can be assigned to the properties (like porosity, permeability, etc.).

f) Hydrus

g) COMSOL

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The Centre for X-ray Tomography (UGCT) is a UGent Expertise Centre which performs research on and with the X-ray micro-CT technique. UGCT also acts as a user facility that offers the use of X-ray CT to researchers of many scientific domains in their research. It was founded in 2006 and is one of the few CT centers in the world that cover the complete CT workflow: physics and instrumentation, data reconstruction and data analysis.

The UGCT is operated by a multi-disciplinary team and is currently an interfaculty collaboration between 3 research groups: the Radiation Physics group, the Pore-Scale Processes in Geomaterials Research group and the Laboratory for Wood Technology

UGCT performs research on laboratory based very high-resolution X-ray tomography and its applications, covering the complete imaging chain. This includes CT scanner development, simulation of the imaging chain, the study of novel imaging techniques, developing tomographic reconstruction methods (including iterative reconstruction techniques and GPU-based algorithms) and 3D image analysis methods. The developed hardware and methodology are used within UGCT for geological and wood technology research.

As a user facility, UGCT offers external research groups and companies access to its unique combination of state-of-the-art in-house developed high-resolution X-ray CT systems and its CT expertise. The flexibility of the systems and the knowledge of the researchers involved allows to perform challenging experiments in various research fields, continuously pushing the limits of what is currently possible with CT.

For more info: www.ugent.be/we/ugct/en

Environmental Micro-CT / EMCT

The Environmental Micro-CT or EMCT system is a rather unique, gantry-based high-resolution setup developed for fast CT scanning and in-situ monitoring. The design of a horizontal gantry allows for the installation of many add-on modules such as flow cells, pressure stages, temperature stages,... in a convenient vertical position without a limitation on tubes and wires. Furthermore, the components are chosen to enable fast and continuous micro-CT scanning at up to 5 full rotations per minute.

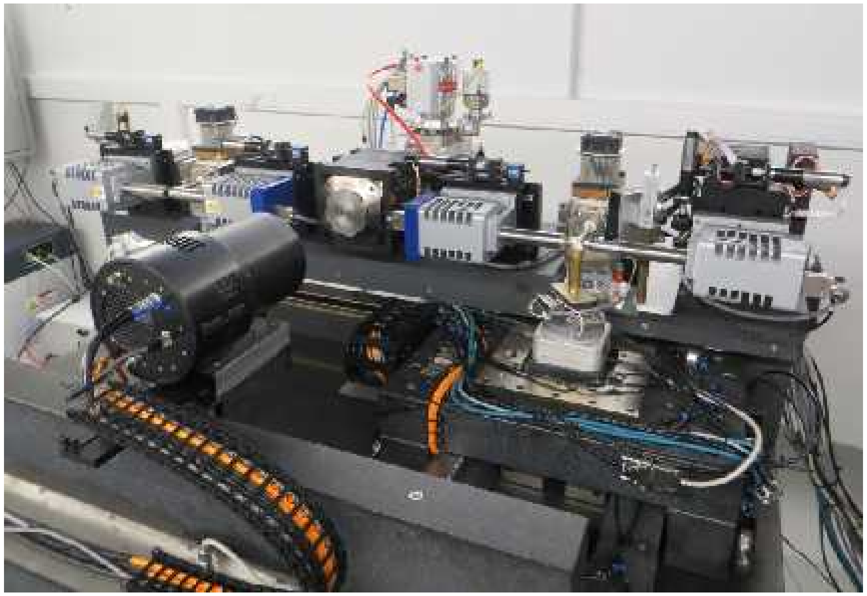
More details can be found in Dierick *et al.*, Nucl. Instr. Meth. Phys. Res. B **324**, 35-40 (2014).
Illustrative examples of fast, in-situ imaging are explained in Bultreys *et al.*, Adv. Water Res. **95**, 341-351 (2014).

The High-Energy CT system Optimized for Research / Hector

The High-Energy CT system Optimized for Research or HECTOR is the workhorse of our systems. It is equipped with a 240 kV X-ray tube from X-RAY WorX, a PerkinElmer 1620 flat-panel detector and a rotation stage able to carry samples up to 80 kg. Mounted on a total of 5 motorized linear stages, this system covers a very wide range of samples with a best achievable spatial resolution of approximately 3 micron and an image resolution of 2048x2048 pixels, which can be extended by tiling both horizontally and vertically. Two additional piezo stages allow for an exact positioning of the sample on the rotation axis.

More details can be found in the dedicated paper by Masschaele *et al*, J. Phys. Conf. Series, **463** (2013).

Herakles



The combined micro-CT - micro-XRF system Herakles combines three scanning stages for extensive sample characterization. One high-resolution CT stage is complemented with two micro-XRF stages, where the three stages are linked by an innovative air-beared positioning system which offers a sub-micron accuracy over the complete setup, necessary for the image correlation.

This system was developed and constructed in collaboration with the X-ray Microspectroscopy and Imaging group of Prof. L. Vincze (XMI) with the financial support of the Hercules Foundation (project AUGE/11/024. 2011).

More details can be found in the dedicated paper by Laforce *et al*, Anal. Chem. **89**, 10617-10624 (2017).

Medusa

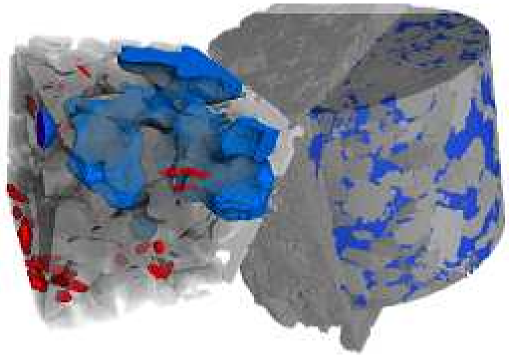
The very high resolution scanner Medusa is the re-designed version of the first UGCT sub-micron CT system, of which a description can be found in Masschaele *et al*. (2007). Similar to Nanowood, it combines a Photonic Science VHR detector with a large-area Varian flat-panel detector to allow for both low-density objects such as biological tissue, and high-density samples such as geomaterials. Both detectors are mounted on motorized linear stages for easy and fast switching and high accuracy. On this stage, additional space is available for experimental testing. The setup also allows for a very long propagation distance of 1.4m, which can be exploited for phase-contrast experiments. The FeinFocus transmission tube allows for a resolution of approximately 0.9 µm and for X-ray targets of different material and thickness.

Nanowood

The extremely versatile multi-resolution X-ray tomography scanner is located at the Laboratory for Wood technology, hence its name. It is equipped with two separate X-ray tubes and two different X-ray detectors to allow for optimal scanning conditions for a very wide range of samples. The open-type Hamamatsu transmission tube is used for very high-resolution CT scans, where a resolution of approximately 0.4 micron can be achieved, whereas the closed-type Hamamatsu directional tube head is used for larger samples. On the detector side, an 11 megapixel Photonic Science VHR CCD camera with a pixel size of approximately 7^2 µm^2 is complemented with a large-area Varian flat-panel detector. Using 7 motorized linear stages, X-ray tube and detector can be switched with a click of a button, and the scanning geometry can be chosen to optimize the detected X-ray flux or optimize the X-ray propagation distance for phase contrast imaging.

More details about this system can be found in Dierick *et al.*, Nucl. Instr. Meth. Phys. Res. B 324, 35-40 (2014).

PProGRess (Pore-scale Processes in Geomaterials Research group) studies the various processes that occur inside the pore space of geomaterials. The group specializes in non-destructive imaging of the 3D structure from the nano- to macro-scale. Our group wants to stretch the limits of real-time imaging of processes in the pore space of geomaterials, in terms of both temporal and spatial resolution.



PProGRess evolved from the “Sedimentary Geology and Engineering Geology” (SGIG) research group, and therefore still holds a lot of expertise in the field of natural building stones and the assessment of their properties. PProGRess researchers are skilled in many traditional research techniques, e.g. optical microscopy, grain size analysis and technological testing of natural building stones.

PProGRess is part of the Ghent University Centre for X-ray Tomography (UGCT) and is member of the department of Geology at Ghent University.

For more info: pprogress.ugent.be

Setup for «CT imaging of fluid flow experiments

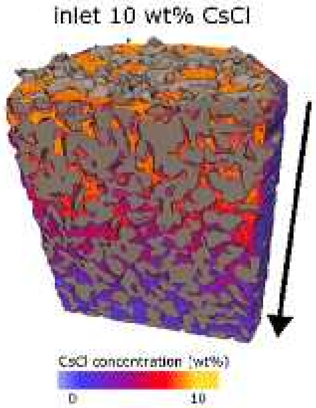
What: Direct observations of flow and transport in permeable rocks at the pore scale

Examples: near real-time imaging of solute and reactive transport, and pore scale physics of multi-phase flow.

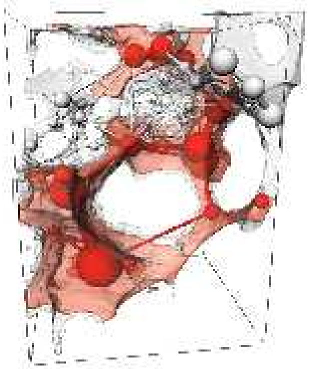
How: Fast X-ray imaging of one or two fluids as they are pumped through a cylindrical rock sample mounted in a custom designed X-ray transparent core holder. The flow can be pressure or rate controlled, with optional inline pressure and salinity measurements. Image based pore-scale properties (e.g. concentration fields, fluid distributions) can then directly be compared to numerical simulations.

Setup

Results and Analysis



The transient solute concentrations in a sintered glass sample imaged using dynamic µCT.



The distribution of a non-wetting phase during imbibition compared to the simulated distribution on a pore-network.

Limits of the method:

Currently the set-up is limited to 50 bar confining pressure, ambient temperatures and sample diameters of 6 mm.

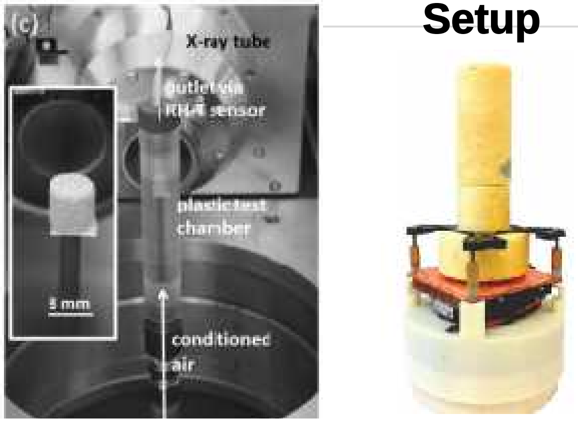
Setup for «CT imaging of weathering experiments

What: Direct observations of fluid transport, crystallization and damage generation in porous materials

Examples: study on damage induced by ice crystals, salt crystal growth, gypsum crust formations

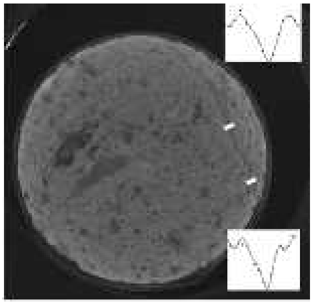
How: Simulation of weathering conditions while performing X-ray imaging is facilitated by the use of custom-made environmental cells. These includes a cell to control temperatures between -20 °C and 100 °C, a cell to control relative humidity (R.H.) and a cell to bring a sample's surface into contact with a solution. X-ray imaging is then used to monitor changes in the solid matrix and/or the pore space.

Setup

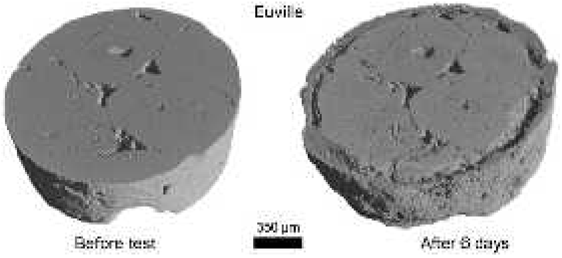


Left: a cell in which the R.H. & T can be maintained. Right: a custom-made cell able to perform freeze-thaw cycles (De Schryver *et al.*, 2016)

Results and Analysis



A slice through a volume taken directly after ice crystallization occurred using dynamic µCT. Ice crystals caused the opening of a crack.



3D renderings of a limestone before and after a weathering test. Degradation of a limestone by formation of a gypsum crust is clearly noticeable.

Limits of the method:

Small sample diameters. The application of a cell reduces the spatial resolution. Only processes slower than 12 seconds can be imaged.

Zeiss Axioscope A1 microscope

What: characterization of mineralogical and textural features in stones

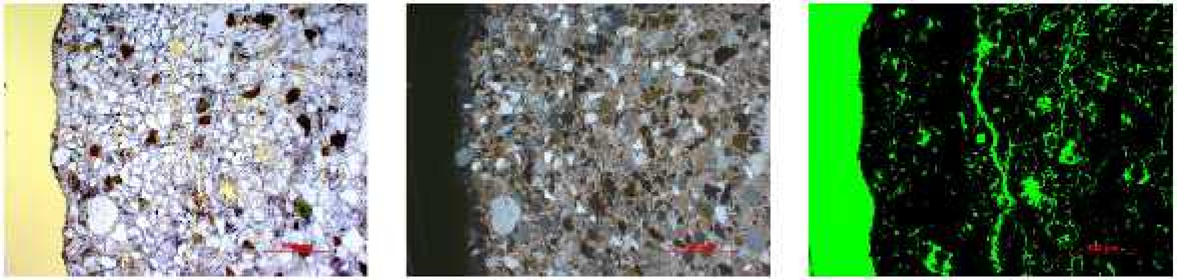
Example: characterization of building stones, evaluation of susceptibility of materials to weathering

How: petrographic thin section analysis under transmitted-light microscope in plane polarized and crossed-polarized light, fluorescence imaging

Setup

Microscope equipped with Axiocam and EpoDye filter.

Results and Analysis



Transmitted light PPL

Transmitted light XPL

Reflected light PPL

Characterization of a laminar black gypsum crust on a sandy limestone (De Kock et al. 2017)

Limits of the method: destructive sample preparation

Spectrophotometer CM-600d Konica Minolta

What: color measurements

Example: analysis color changes building stones after weathering experiments

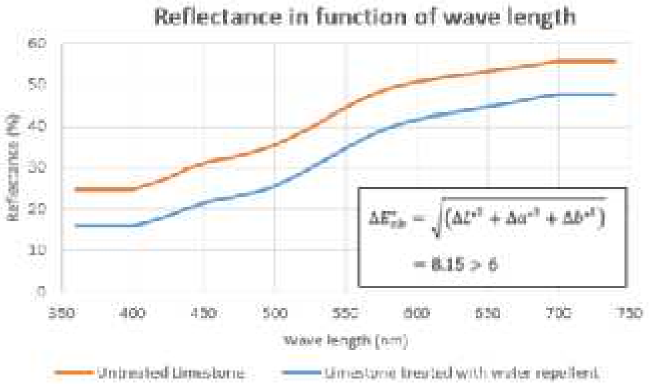
How: the spectrophotometer measures a light beam's intensity as a function of its color (wavelength), the device can be used in both laboratory and the field

Setup

Specifications:

- Wavelength range: 400 nm to 700 nm (10 nm-interval)
- Measuring aperture: ø 8 mm
- Color spaces: L*a*b*, L*C*h, Hunter Lab, Yxy, XYZ, Munsell
- Displayed data: spectral values, colorimetric values, color difference values, …

Results and Analysis



Assessment of color changes of a limestone after treatment with a water repellent. A color difference (ΔE^{*ab}) higher than 6 is visible for the human eye.

Limits of the method:

Tescan Scanning Electron Microscope (SEM)

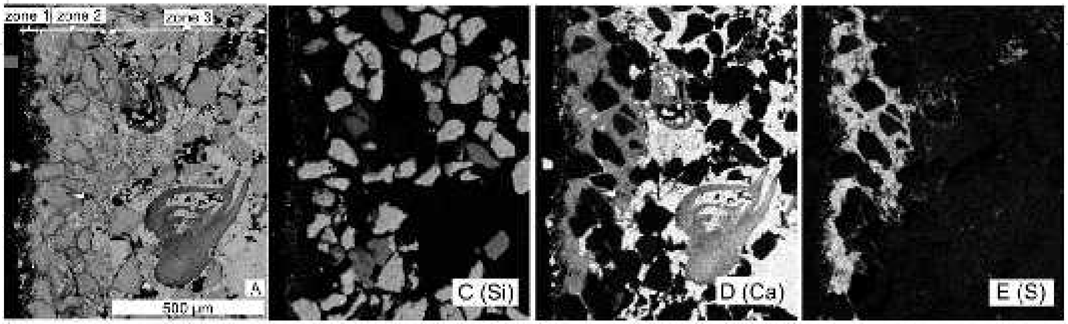
What: imaging (SE, BSE, CL), EDS analysis and Integrated Mineral Analyzer

Example: characterization of weathering features (e.g. gypsum crusts)

How: focused beam of electrons scans surface of sample, intensity of detected signal produces image containing information of surface topography (SE) and chemical composition (BSE, EDS) of the sample.

Setup

Results and Analysis



(A) SEM-BSE image of gypsum crust (zone 1: opaque upper layer, zone 2: gypsum crystallization layer, zone 3: sound stone); (C-E) SEM-EDS mapping of Si, Ca, S (De Kock et al. 2017)

Limits of the method: High vacuum: carbon/gold coating necessary for non-conductive samples, low vacuum: for “wet” samples

FLIR Heat Camera A655sc 45°

What: thermal imaging and measurements

Example: monitoring of temperature changes during freeze-thaw cycles, salt crystallization

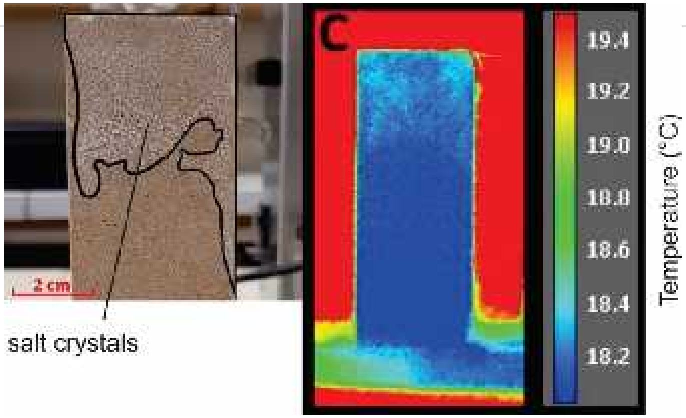
How: camera detects long range IR emission. The quantity of emitted IR radiation is related to the temperature of the visualized subject.

Setup

Specifications:

- 640 x 480 pixels image resolution
- Standard camera calibration ranges: -40°C to 150°C, 100°C to 650°C
- Includes high temperature option (300-2000°C)
- IR lens, f=13.1 mm (FOV=45°)

Results and Analysis



Left: a photograph of a prismatic limestone sample after a salt crystallization experiment. Right: an IR thermography between 18 and 19.5 °C of the same sample during the crystallization process. This is visible as warmer temperatures occur at the top of the prism.

Limits of the method: needs connection to laptop and power source. Surficial temperatures may be influenced by exterior processes

Delft University of Technology

Delft University of Technology (Dutch: Technische Universiteit Delft) also known as TU Delft, is the largest and oldest Dutch public technological university, located in Delft, Netherlands. With eight faculties and numerous research institutes, it hosts over 19,000 students (undergraduate and postgraduate), more than 2,900 scientists, and more than 2,100 support and management staff.

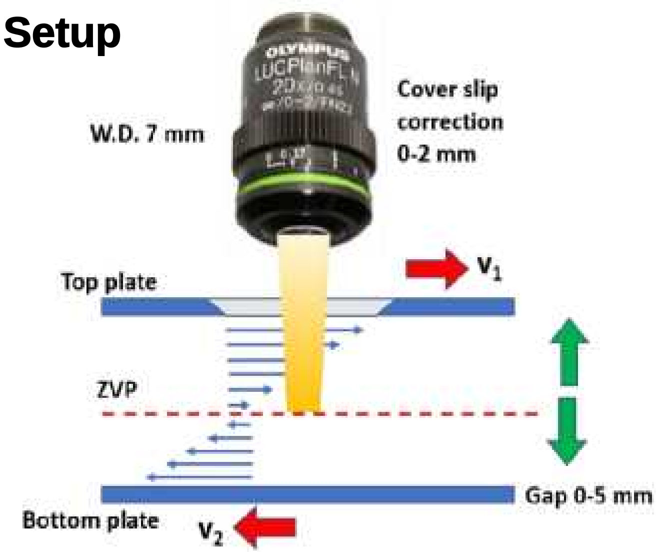
Rheo-optics – suspensions under shear

What: Visualize the behavior of a suspension under shear

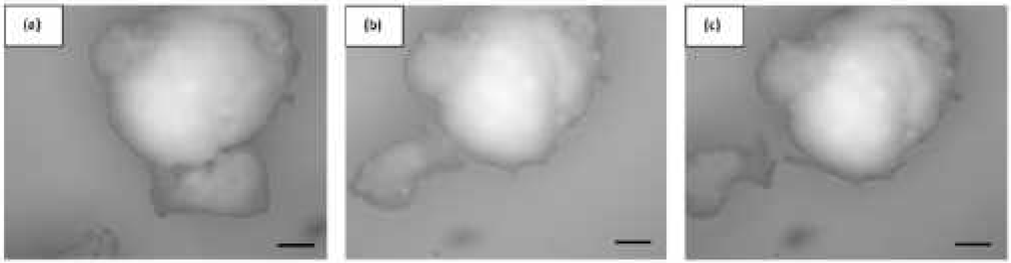
Example: fluid mud samples, any colloidal suspensions, pastes

How: Video microscopy coupled with shearing cell

Setup



Results and Analysis



Images of cationic polyelectrolyte-based kaolin suspensions subjected to oscillation at (a) t = 0 s, (b) t = 3 s, (c) t = 5 s; Gap width = 100 lm; f = 1 Hz; A = 0.4 mm. Sequence of images shows the break-up of flocs. Scale bar represents 70 mm.

Limits of the method: the shear rate is controlled, but the stress cannot be measured. The device should be used in combination with a standard rheometer to assess the shear-stress relationship.

Rheology – flow properties

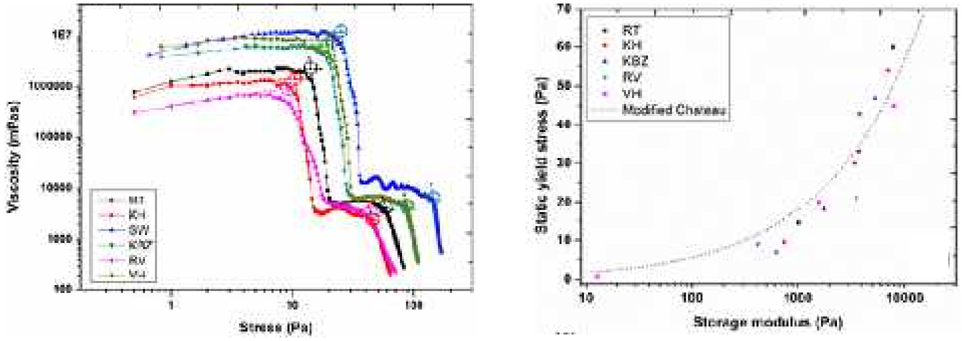
What: Measurement of yield stress, viscosity, storage and loss modulus

Example: fluids, suspensions, pastes

How: a steady or oscillatory shear or stress is imposed on the sample. The corresponding stress or shear is measured.

Setup

Results and Analysis



Left: viscosity as function of stress and right: yield stresses as a function of storage modulus. the data is for mud sediments from different locations. The dotted lines represent the model fitting

Limits of the method: wall slip and system inertia can hamper the measurements

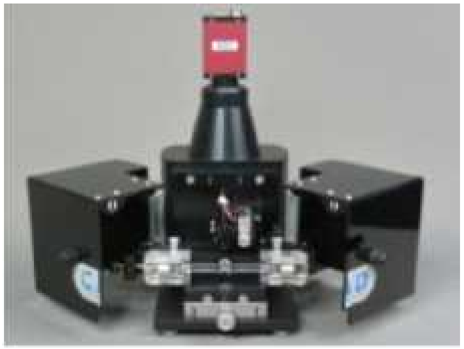
Video electrophoresis – zeta potential

What: assess electric surface properties of nano or micrometric particles

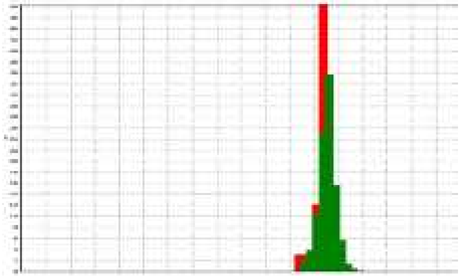
Example: dilute colloidal suspensions

How: record the velocity of nano or micrometric particles as function of applied electric field

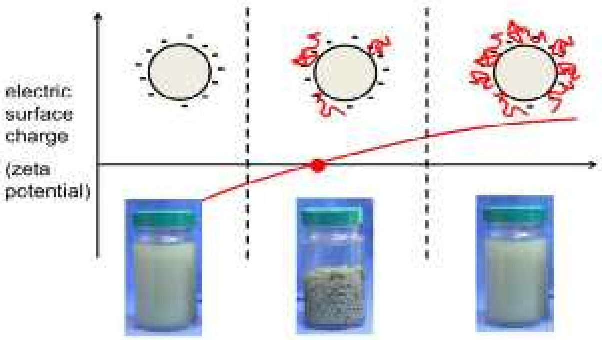
Setup



Results and Analysis



Particle number distribution (% particles) as function of zeta potential. The zeta potential is proportional to the recorded electrophoretic velocity.



Zeta potential as function of flocculant dosage. Depending on the zeta potential, stable or unstable suspensions are created. A zero electrophoretic mobility implies a zero zeta potential.

Limits of the method: the suspensions should be diluted enough for the video to record and the software to track properly the particles.

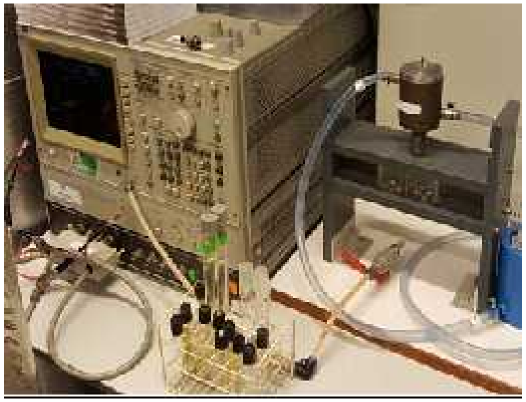
Dielectric Spectroscopy

What: assess the main polarization mechanisms in solutions and suspensions

Example: ionic solutions, liquids, colloidal suspensions

How: record the complex impedance of the fluid as function of frequency. From the impedance the dielectric permittivity and the conductivity of the fluid can be calculated.

Setup

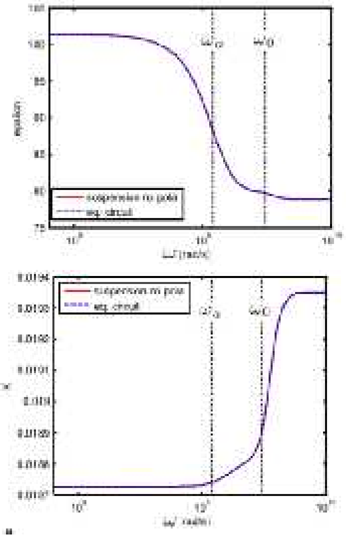


impedance meter and home-made cell

Results and Analysis

Relative permittivity (epsilon) and conductivity (K) as function of frequency for a suspension of 100 nm colloidal spheres in a 1 mM NaCl solution. Two relaxation frequencies are visible associated to the particle (ω_a) and the double layer (ω_0) sizes.

The curves are theoretical predictions for illustration. The measured curves show additional effects due to so-called electrode polarization at low frequencies. This polarization can be accounted for to “clean” the data.



Limits of the method: the measurements are very sensitive to the state of the electrodes and the cell geometry. This should be accounted for to derive useful results.

Electric birefringence

What: assess nanoscopic particles’ relaxation mechanisms or collective effects

Example: dilute suspensions of nanoscopic particles

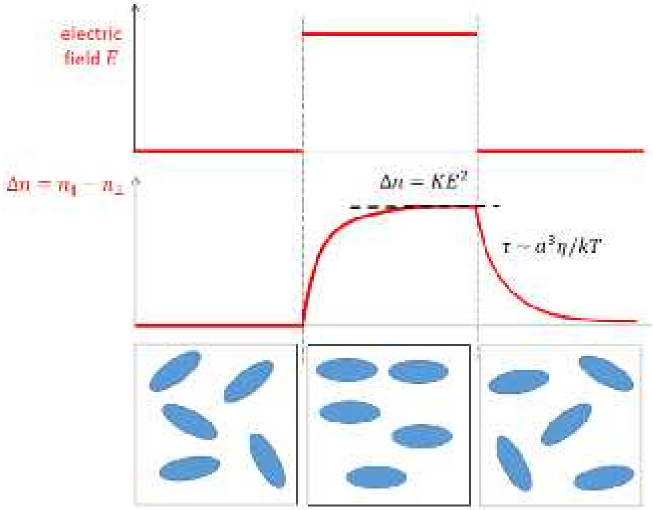
How: study the change in birefringence induced by applying an electric field on a suspension

Setup

The phase shift between the parallel and perpendicular components of the laser light with respect to the applied electric field is proportional to the birefringence Δn.

Results and Analysis

Schematic illustration of the principle: the electric field induces the alignment of anisotropic particles in the electric field direction. This leads to the measured birefringence Δn which is proportional to the electric field squared. The relaxation time gives information about the particle size.



Limits of the method: only dilute suspensions can be studied as sufficient light should reach the photodetector.

Shell Global Solutions International B.V.

Technology development is at the heart of what the Shell Technology Center Amsterdam does. Our researchers are constantly looking for ways to improve our products and processes and to invest in more efficient and cleaner technologies. This covers activities ranging from research in the domain of catalysis and processes related to refineries to research in porous rocks. At the Shell Technology Center in Amsterdam a wide range of experimental facilities and experimental techniques are used.

Contact person: steffen.berg@shell.com

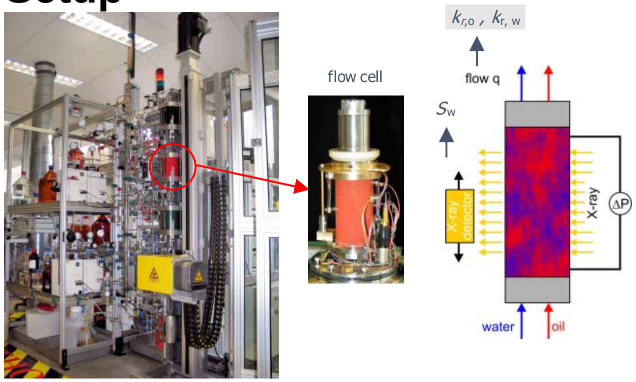
Conventional and Special Core Analysis

What: characterization of rock samples in terms of flow properties

Example: porosity, permeability, relative permeability and capillary pressure (for two-phase flow)

How: flow experiments on cylindrical rock samples ("cores")

Setup

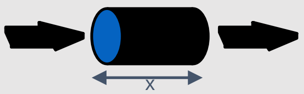


steady state flow measurements: inject oil/water mixture until oil and water saturations S_o, S_w and P_c stay constant, measure pressure drop ΔP, flow rates q → relative permeabilities $k_{r,o}$, $k_{r,w}$

Results and Analysis

Single-phase flow
absolute permeability

water



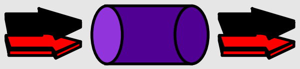
Darcy's law

$$q = \frac{A.K}{\mu}\left(\frac{dP}{dx}\right)$$

flux q, cross section A, viscosity μ, pressure P, permeability K

multiphase flow
Relative permeability

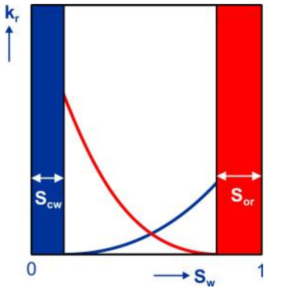
water, oil



$$q_o = \frac{A.K}{\mu_o}\left(\frac{dP_o}{dx}\right).k_{ro}(S_0)$$

$$q_w = \frac{A.K}{\mu_w}\left(\frac{dP_w}{dx}\right).k_{rw}(S_w)$$

Relative permeability k_r



water saturation S_w

Limits of the method: permeability in the range of 1 mD – 2 Darcy, rock needs to be consolidated and needs to be homogeneous. Representative wetting conditions need to be restored by adequate cleaning and ageing with e.g. crude oil.

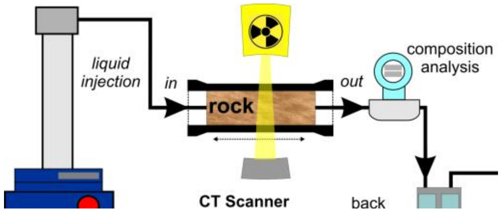
Imaging of Flow Experiments by CT

What: Imaging of fluid distributions by X-ray computed tomography, from a 5 cm - 1 m length scale.

Example: imaging flow front instabilities or formation of wormholes during CO_2 sequestration

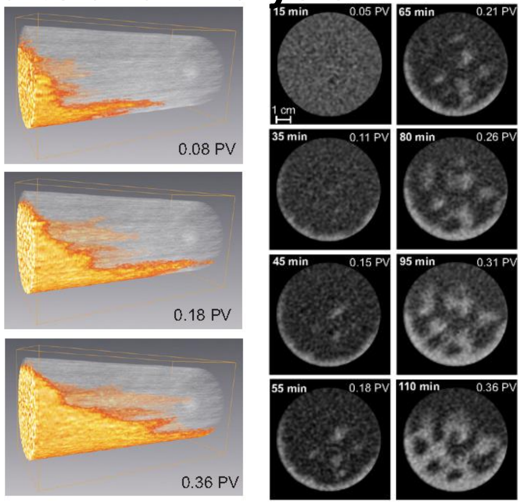
How: conducting core flooding experiments in medical or industrial CT scanners

Setup

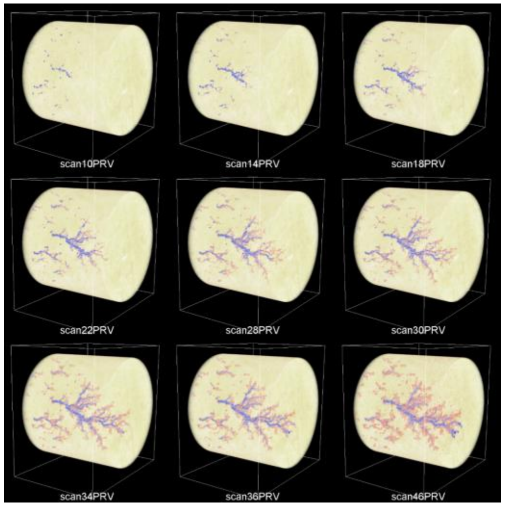


Results and Analysis

Example of (miscible) viscous fingering



Example of wormhole formation in carbonate rock



Limits of the method: for 3D visualization sufficient X-ray contrast between fluids is needed, e.g. by doping one of the fluids with a contrast agent.

Accurate Numerical Resolution of reacting Multi-phase Flow in Fixed and Moving Beds

What: Thermal processing industries, renewable energy e.g. biomass and waste, food and pharmaceutical industries, additive manufacturing, agriculture and its machinery, mining

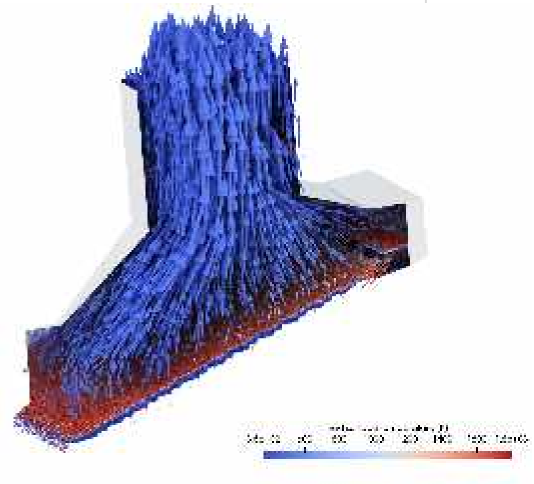
How: Coupling of discrete (DEM) and continuous (CFD) numerical methods through heat, mass and momentum transfer

Setup

Advanced XDEM simulation technologies are available on demand including professional support

Results and Analysis

- Detailed analysis of predicted results allows unveiling the underlying physics and chemistry
- extended understanding of underlying processes enables improved and optimised design and operation for best performance



Limits of the method:

- available computational resources
- requires reliable/validated material properties and reaction kinetics