جامعة الملك عبدالله للعلوم والتقنية
King Abdullah University of Science and Technology

Droplet size distribution measurement of emulsified oil in water: application of optical and electron microscopy techniques

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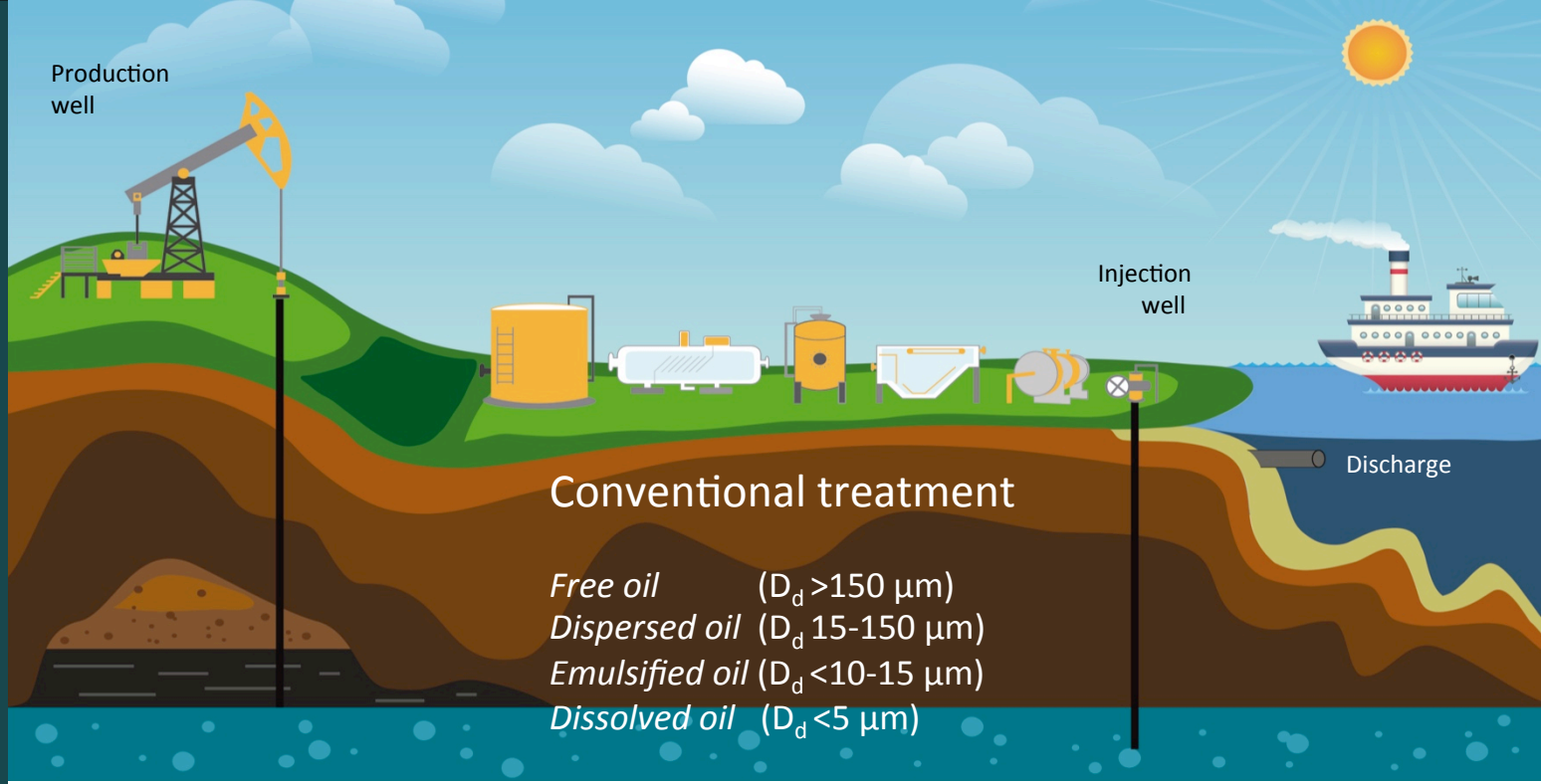
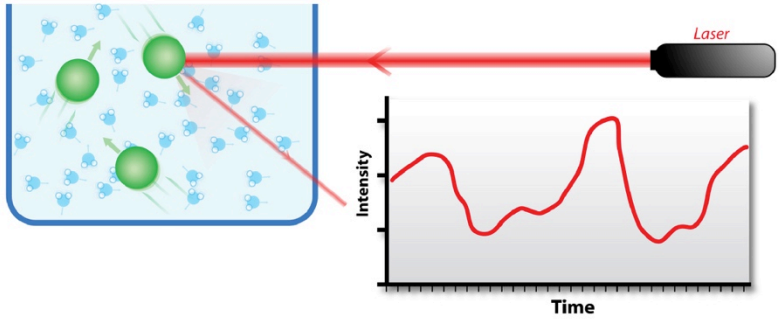
Produced water treatment



Droplet size distribution (DSD) is a key parameter in design and evaluation of treatment strategies for O/W emulsions

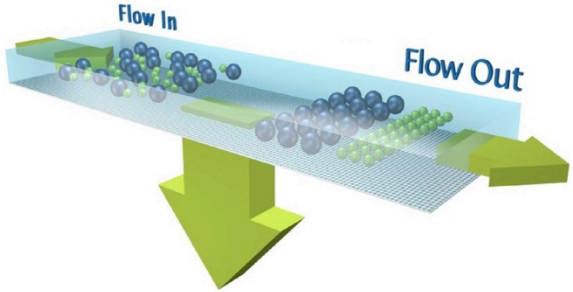
DSD methods

Dynamic light scattering, DLS



Near infrared reflectance

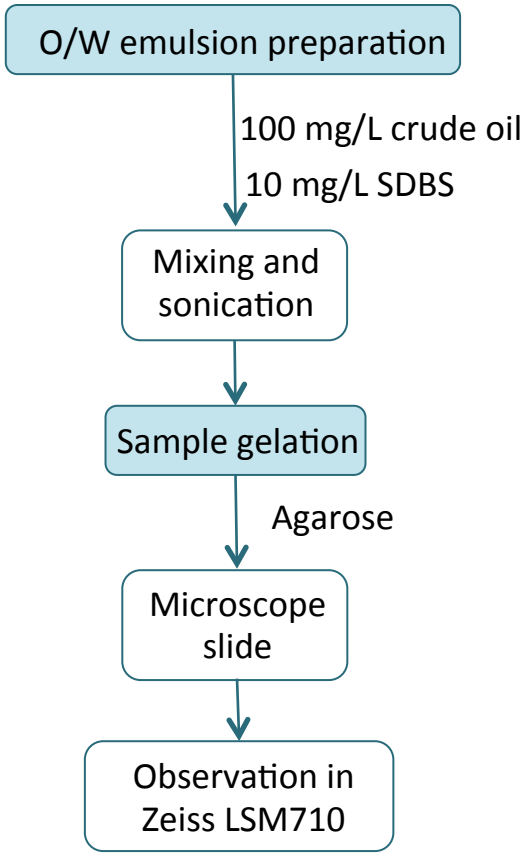
Nuclear magnetic resonance



Field flow fractionation

Direct visualization by optical and/or electron microscopy can provide more accurate determination of the DSD

Optical microscopy: Droplets > 0.3µm



Crude oil droplets can be identified due to the intense fluorescence of its aromatic hydrocarbons

Epifluorescence

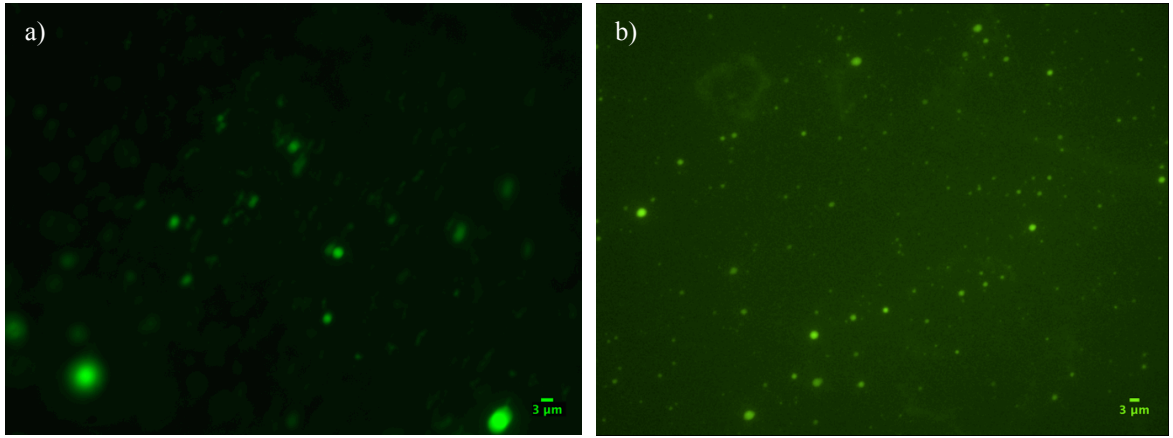
GFP excitation: 485 nm
Emission: 450-485 nm

CLSM

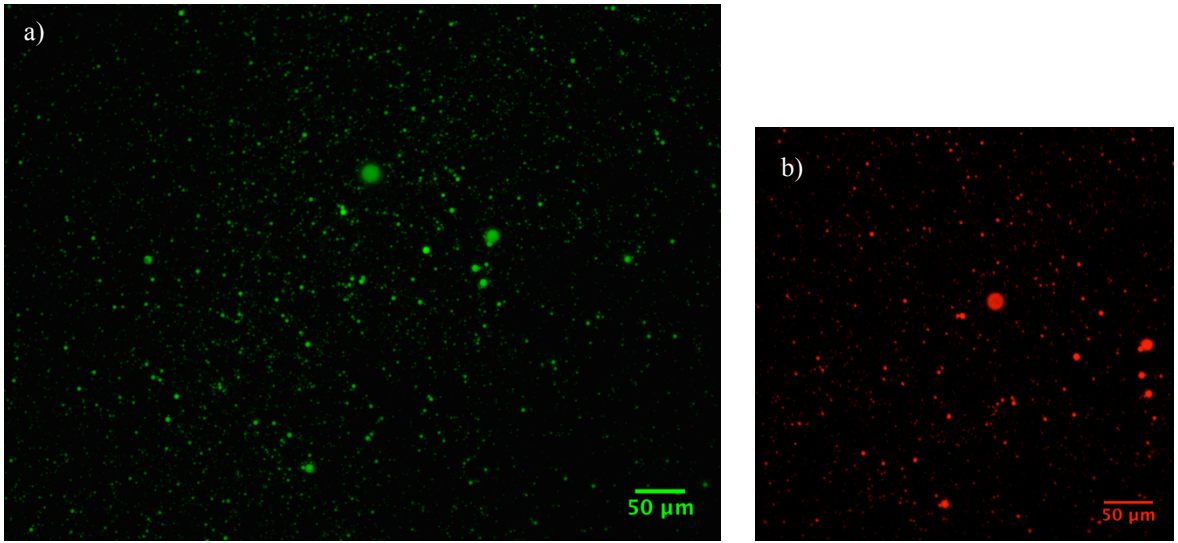
Laser 514 nm
Emission 530-725 nm

Optical microscopy

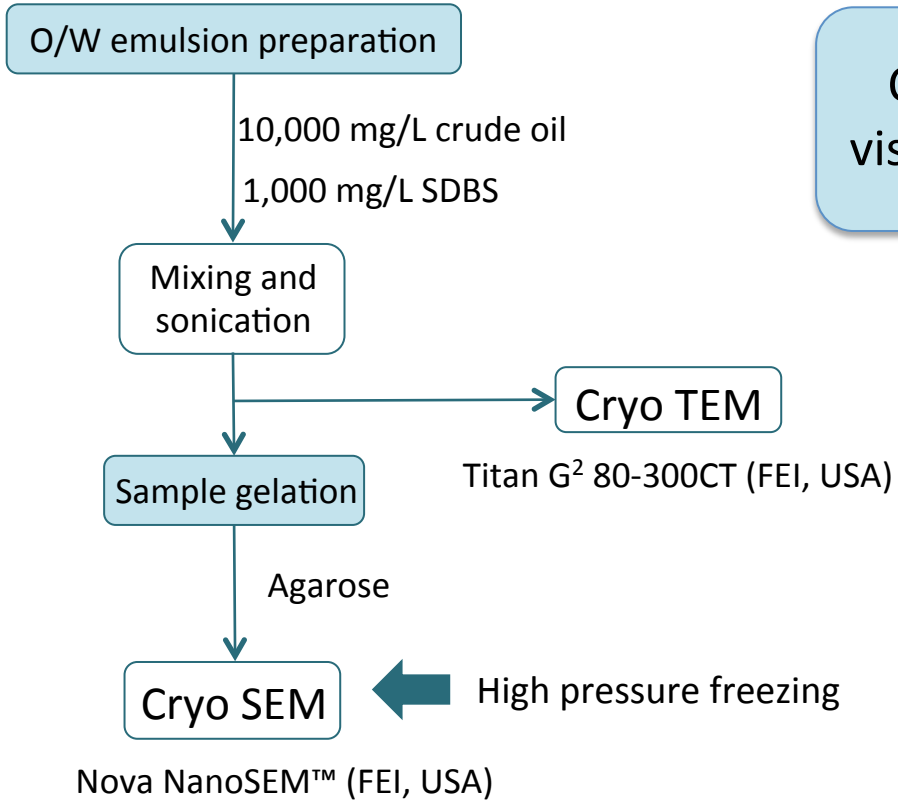
Sample gelation (100X)



Epifluorescence vs. CLSM (20X)



Electron microscopy: Droplets < 0.3µm



Cryo SEM and Cryo TEM were used for visualization of droplets in the nano range

Successful vitrification allows the fixation of constituents without introducing significant alterations

Electron microscopy

Cryo SEM HPF

Cryo TEM

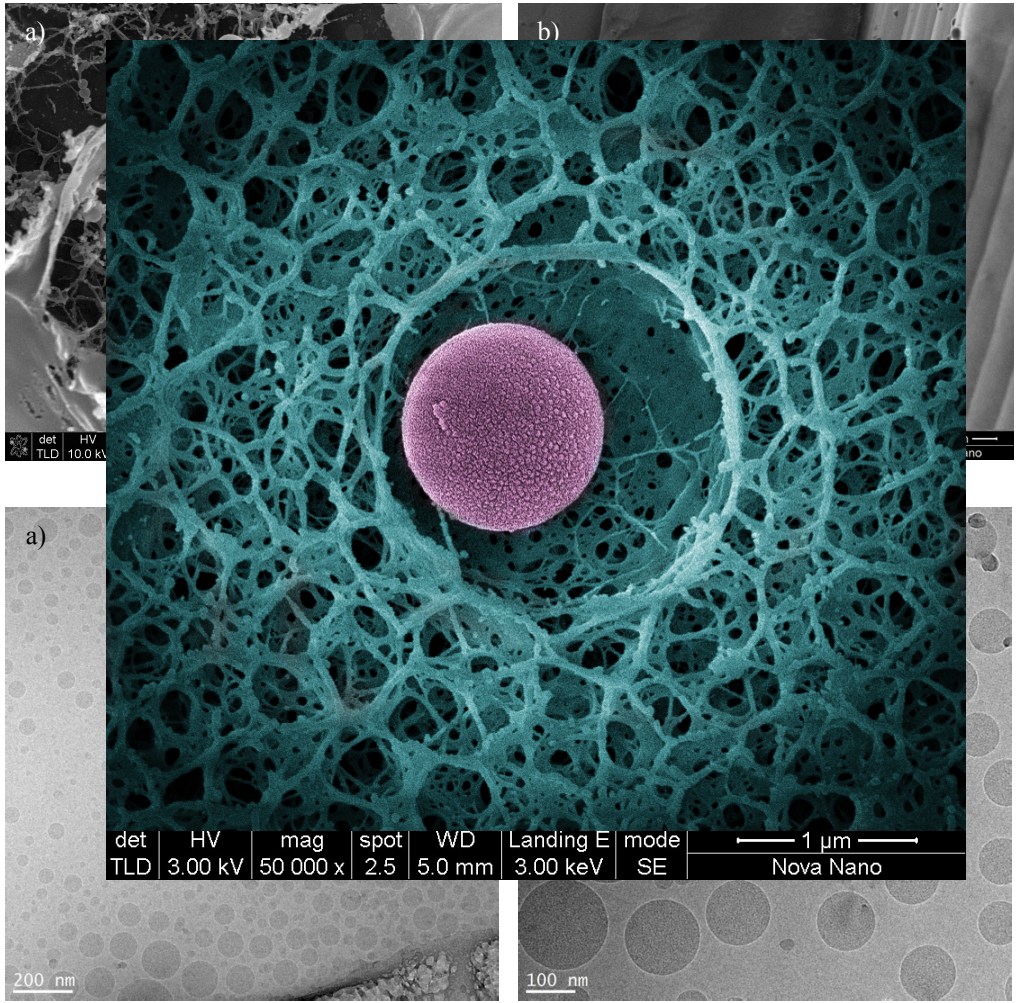


Image post-processing

Image post-processing Image J

Image enhancement filtering

Morphological segmentation

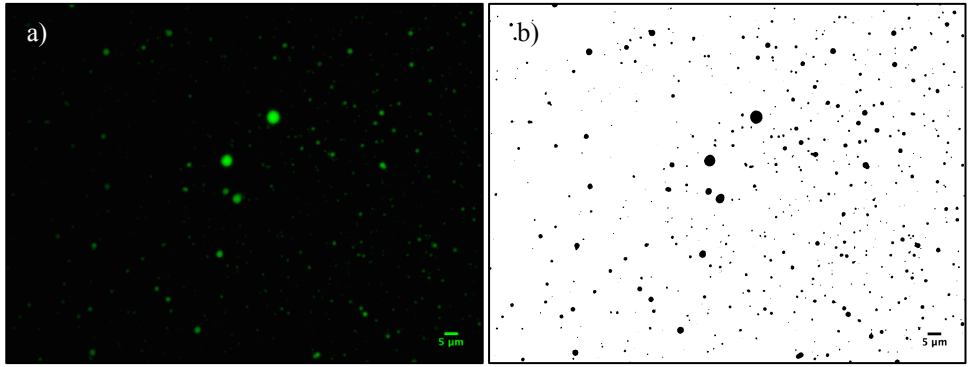
- Subtract background
- Bandpass filter

Calibration and measuring

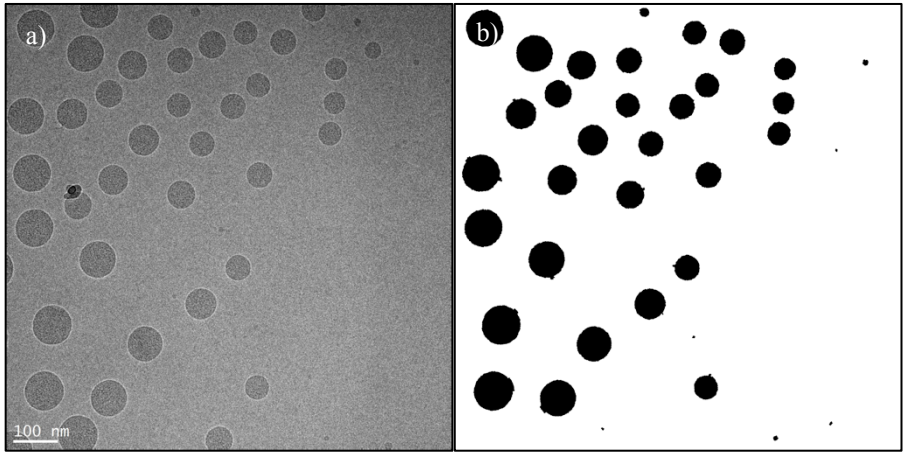
- Watershed
- Circularity criteria

Statistical Analysis

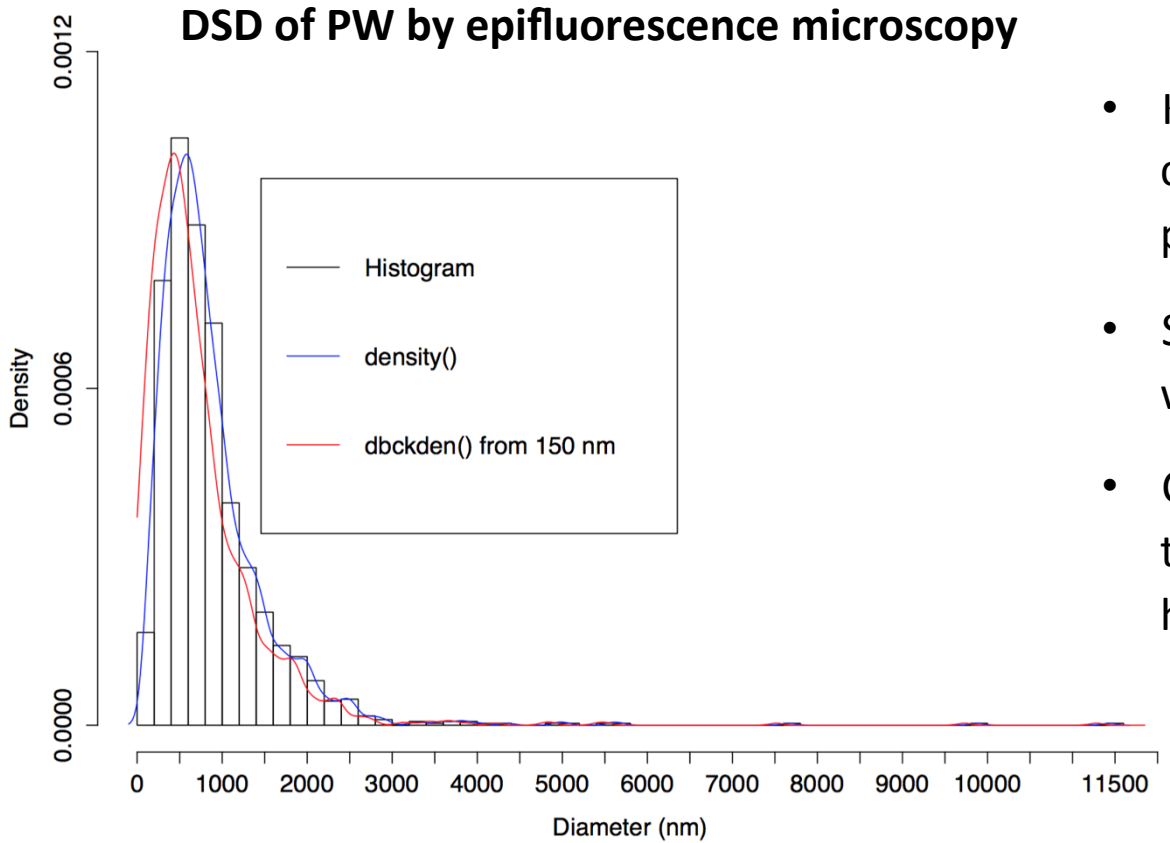
Epifluorescence Microscopy



Cryo TEM



Statistical Analysis



- Kernel density function depends crucially on bandwidth smoothing process
- Sheaher-Jones bandwidth method was selected
- Correction of DSD was applied as the epifluorescence microscopy has a detection limit of 150 nm

Take home messages

- Sample gelation proved to be useful as agarose worked as structural support of oil droplets.
- Epifluorescence microscopy method for determining DSD of O/W emulsions was established and validated, allowing the detection of droplets as low as 150 nm.
- Cryo-SEM is useful for submicron oil droplets visualization but not for DSD analysis.
- Cryo-TEM is useful for determining DSD of nano scale droplets. However several images should be taken to have statistically significant amount of droplets.

Through Inspiration, Discovery

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Thank you