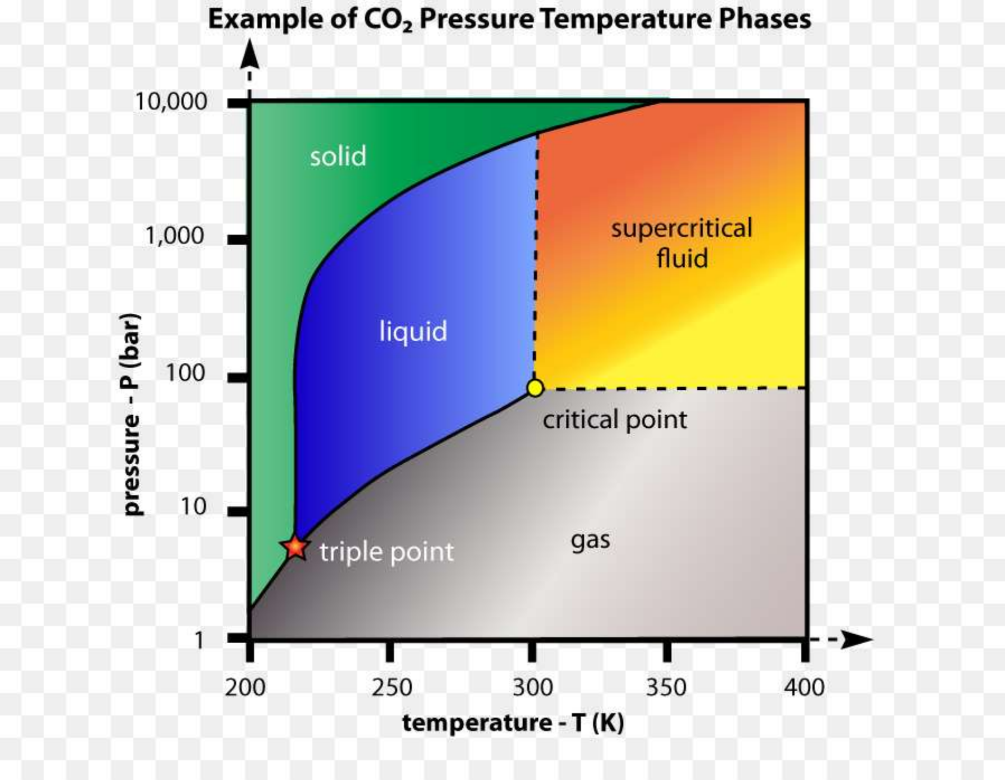
Critical point drying (CPD)

Marvin Wattenbach



UNIVERSITÄT SIEGEN

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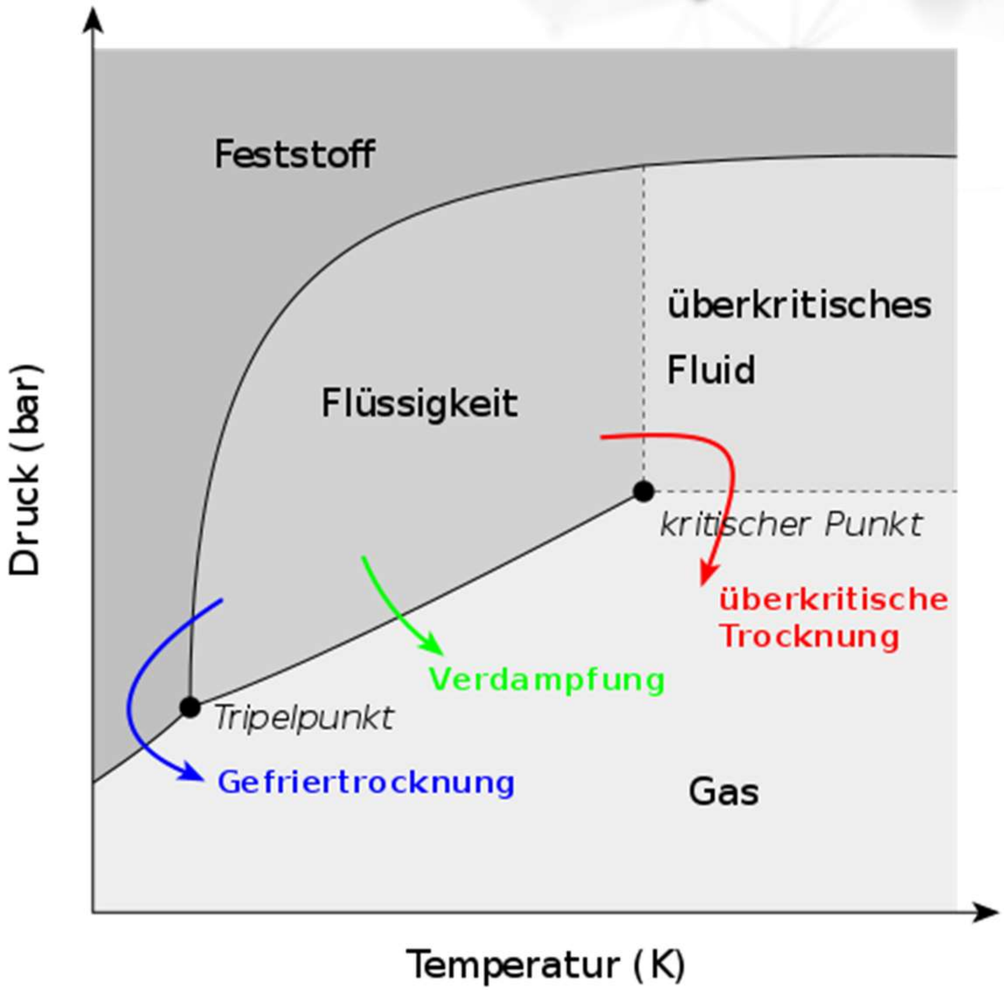
Outlines

- Introduction

- Biological Samples
  - Butterfly Wings

- Non-biological Samples
  - Palladium nanostructure
  - Titania aerogels

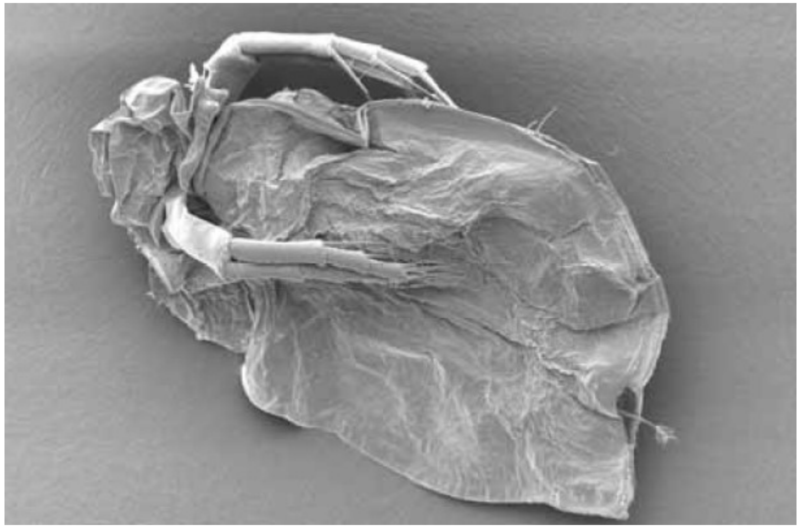
Introduction



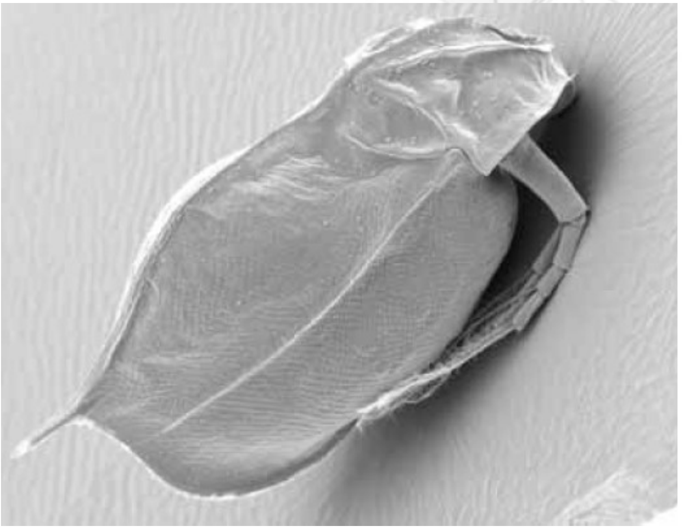
[https://de.wikipedia.org/wiki/%C3%9Cberkritische_Trocknung]

Leica- brief introduction to critical point drying

Used for preparation of biological samples without damages in cells



Air dried water flea



Critical point dried water flea

[https://www.leica-microsystems.com/science-lab/brief-introduction-to-critical-point-drying/]

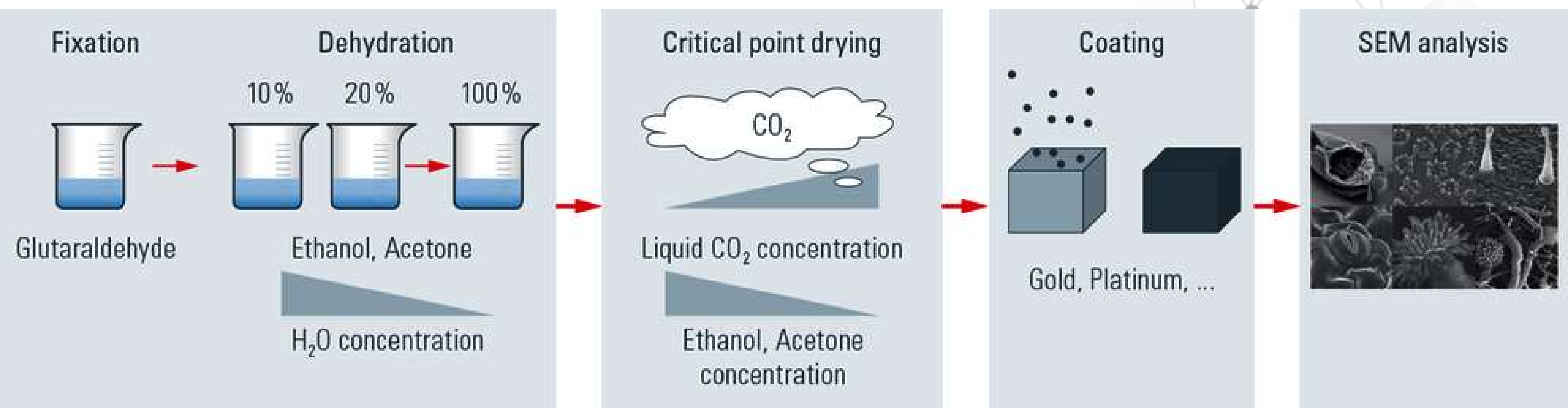
Leica- brief introduction to critical point drying

- CP:
  - Water: 374°C; 229 bar
  - Ethanol:241°C; 60 bar
  - Acetone:235°C; 46 bar
  - Liquid CO2: 31°C; 74 bar

- Replace water by exchange fliuds (ethanol / acetone)
- Exchange fliud with liquid CO2

[https://www.leica-microsystems.com/science-lab/brief-introduction-to-critical-point-drying/]

Leica- brief introduction to critical point drying



Schamatic representation of CPD

[https://www.leica-microsystems.com/science-lab/brief-introduction-to-critical-point-drying/]

Leica- brief introduction to critical point drying

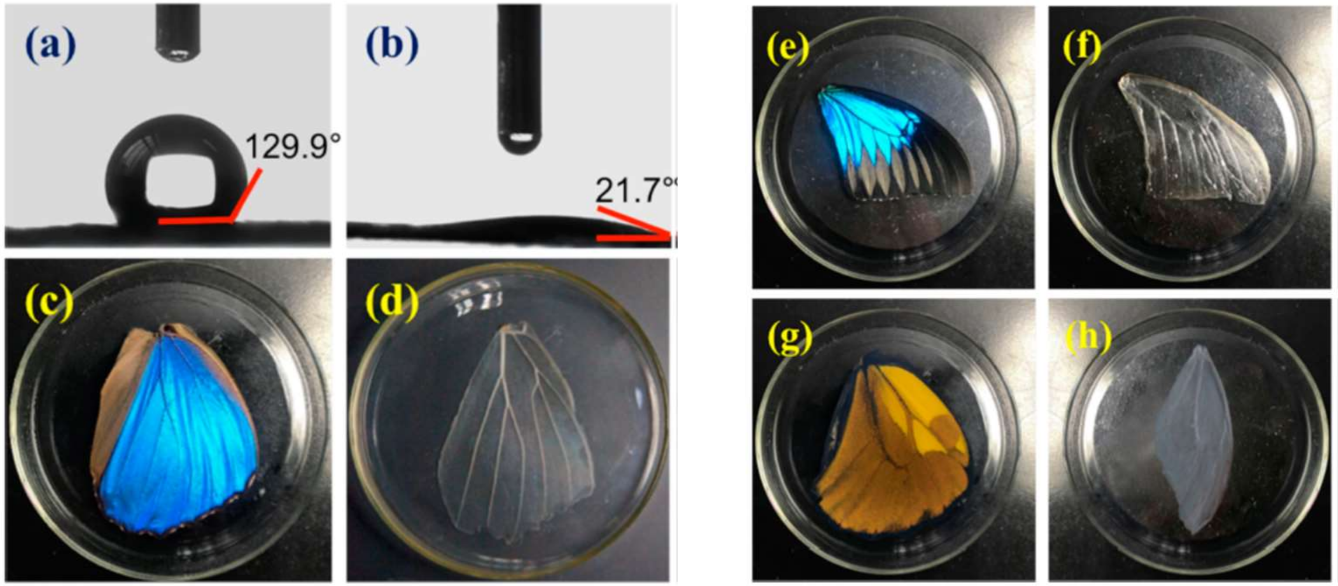
Critical point dryer from Leica

[https://www.leica-microsystems.com/science-lab/brief-introduction-to-critical-point-drying/]

Chitin-based anisotropic Nanostructures of Butterfly Wings for Regulation Cells Orientation

- Use Butterfly wings used as modern alternative cell culturing
- Many applications for cell alignment (embryonic, proliferation, wound healing…)

- Plasma cleaning makes them hydrophilic -> hydroxyl generated on the wings
- Cleaned with HCl an NaOH

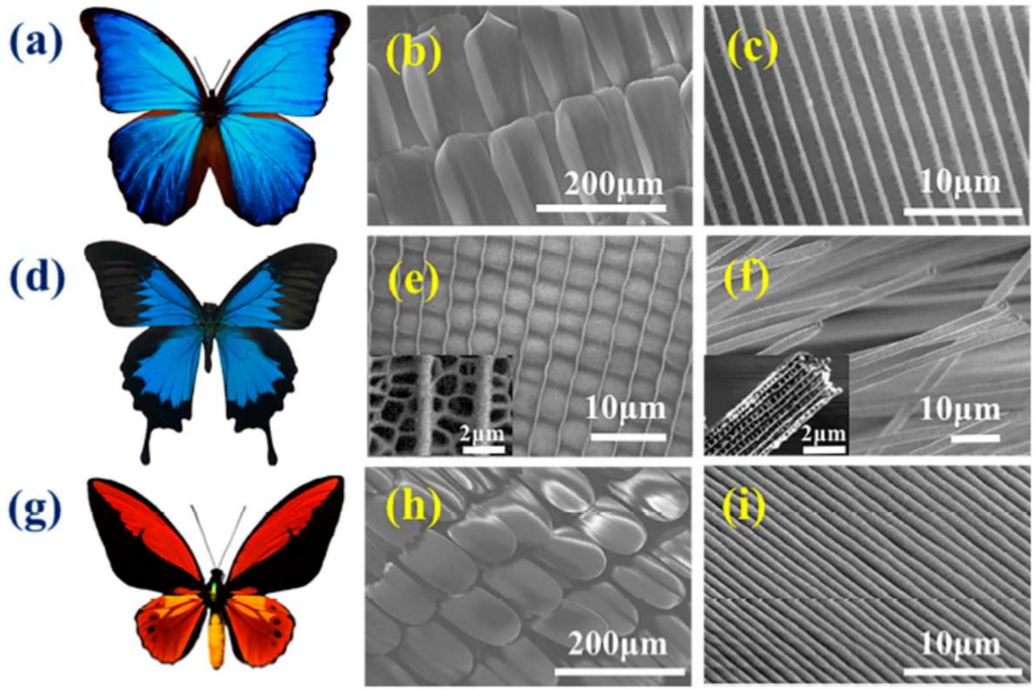


(a) before plasma cleaning
(b) after plasma cleaning
(c,e,g) before cleaning
(d,f,h) after cleaning

[doi:10.3390/polym9090386]

Chitin-based anisotropic Nanostructures of Butterfly Wings for Regulation Cells Orientation

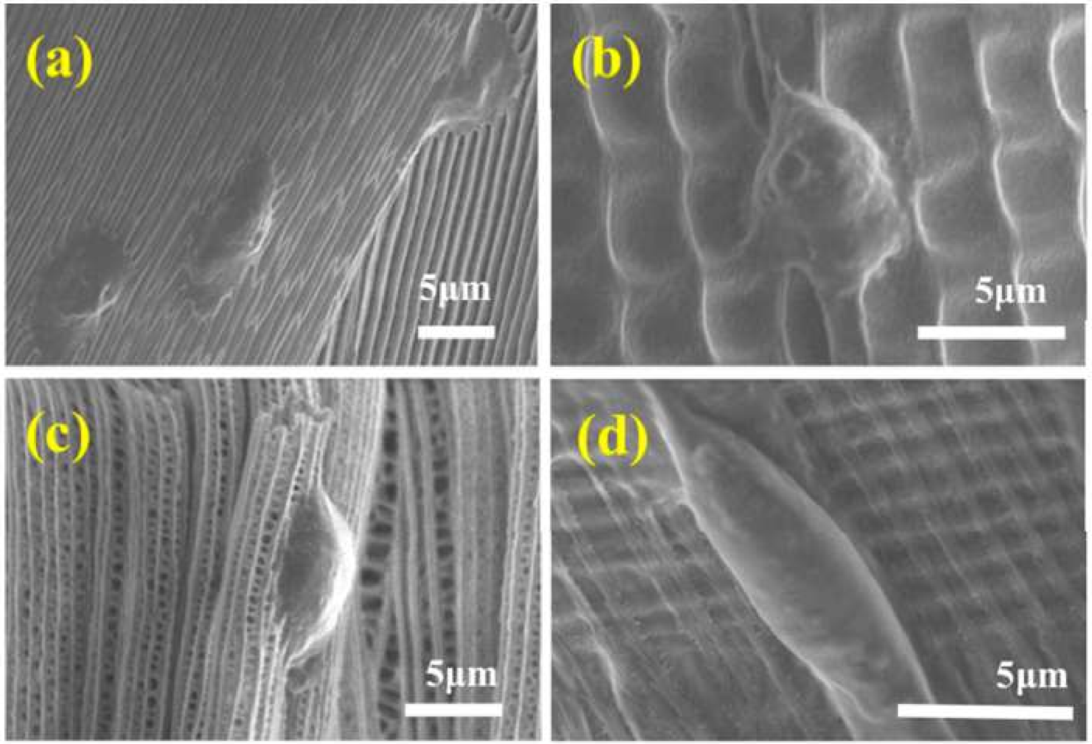
- SEM preparation: 3% of glutaraldehyde for 24 h at 20°C (fixing)
- Dehydrated using a graded series of ethanol (25%, 50%, 70%, 80%, 90%, 95%, and 100%),
- Dried using CO_2 CPD
- Sputter-coated with gold



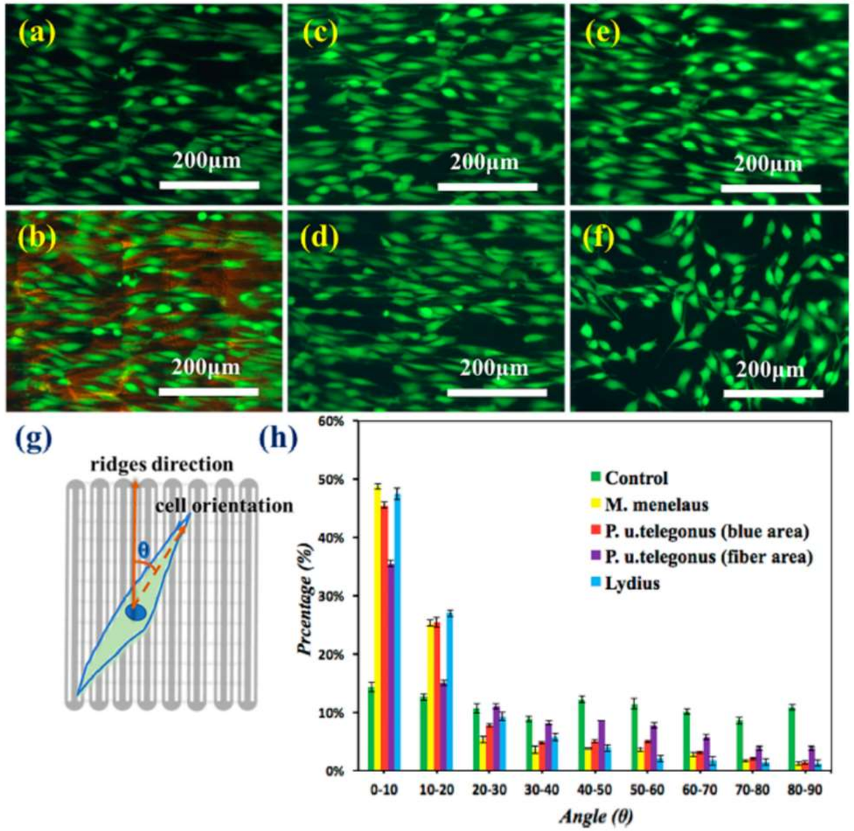
(b),(c) belongs to (a)
(e) in the blue region of (d), (f) in the black region of (d)
(h),(i) belongs to (g) all before cell culturing

[doi:10.3390/polym9090386]

Chitin-based anisotropic Nanostructures of Butterfly Wings for Regulation Cells Orientation



SEM images after cell culturing (a) on blue butterfly, (b) in the blue regions and (c) on the fiber regions at the black and blue butterfly and (d) on the black an red butterfly



(a-e) butterfly wings, (f) culture dish as control, (g) schematic diagram of the orientation angle, (h) distribution of orientation angle of cells

[doi:10.3390/polym9090386]

Non-biological CPD

Palladium nanostructure for hydrogen storage, gas sensing applications and catalytic effects:

- Problems: the formation of nanostructure for large surface area and high catalytic performance

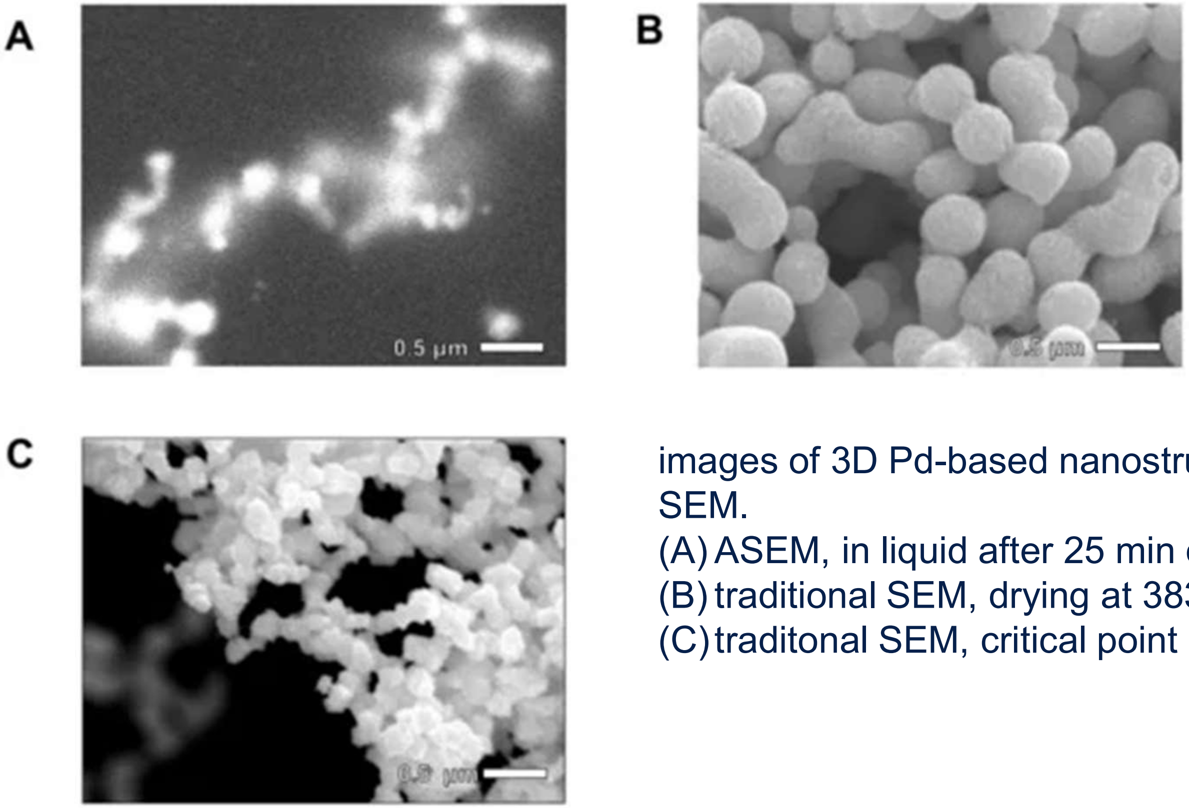
Comparison of lyophilization and critical point drying for **titania aerogels:**

- Used for (photo-)catalysts, pigments, components of batteries and other technically important materials

Palladium nanostructure

Syntheses of Palladium-based nanorings:

- Synthesis of Pd-based nanostructure dissolved in dimethyl sulfoxide (DMSO)



images of 3D Pd-based nanostructures taken by ASEM and a traditional SEM.

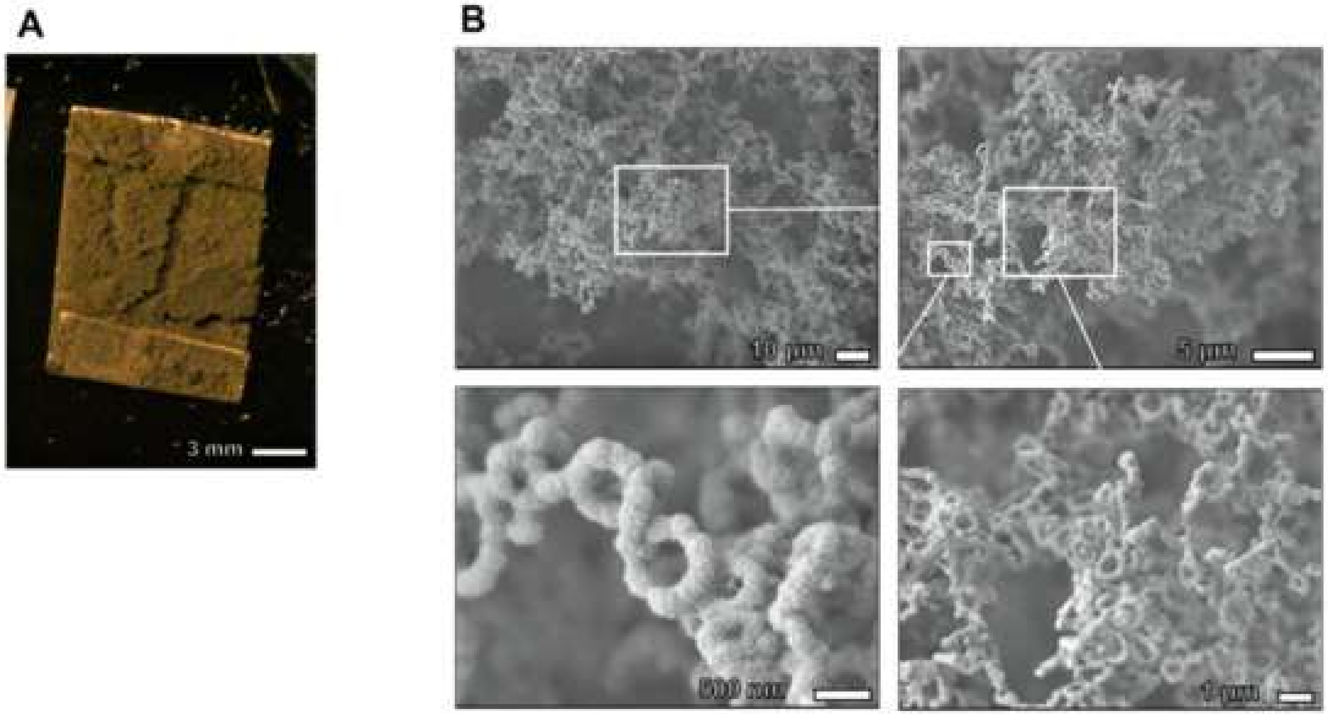
(A) ASEM, in liquid after 25 min of mixing
(B) traditional SEM, drying at 383 K for 24 h
(C) traditonal SEM, critical point drying

[doi:10.3390/ijms21093271]

Palladium nanostructure

Syntheses of Palladium-based nanorings:

- Nanostructured 3D networks by mixing Pd-dissolved DMSO with 2.0 M citric acid aqueous solution -> followed by CPD
- CPD used to avoid surface tension in the drying



fabrication of 3D nanostructure using critical point drying method.
(A) optical microscope image
(B) SEM images of Pd 3D nanostructure

[doi:10.3390/ijms21093271]

Titania aerogels

Preparation of titania aerogel from $TiOSO_4 \cdot nH_2O$:

- Titanium (IV) oxysulfate mixes into water at 35°C -> cooled to 0°C -> added ammonia until pH = 8 and white precipitate was formed
- Precipitate was filtered, washed transferred into beaker and resuspended into water –> with added hydrogen peroxide reduced pH = 3-4 -> suspension turned cloudy yellow
- 1h stirring, suspension became transparent yellow -> rest for a couple of days at room temperature
- Titania aerogels were prepared from the yellow suspension by lyophilization and critical point drying.

[DOI 10.1515/pac-2016-1031]

Titania aerogels

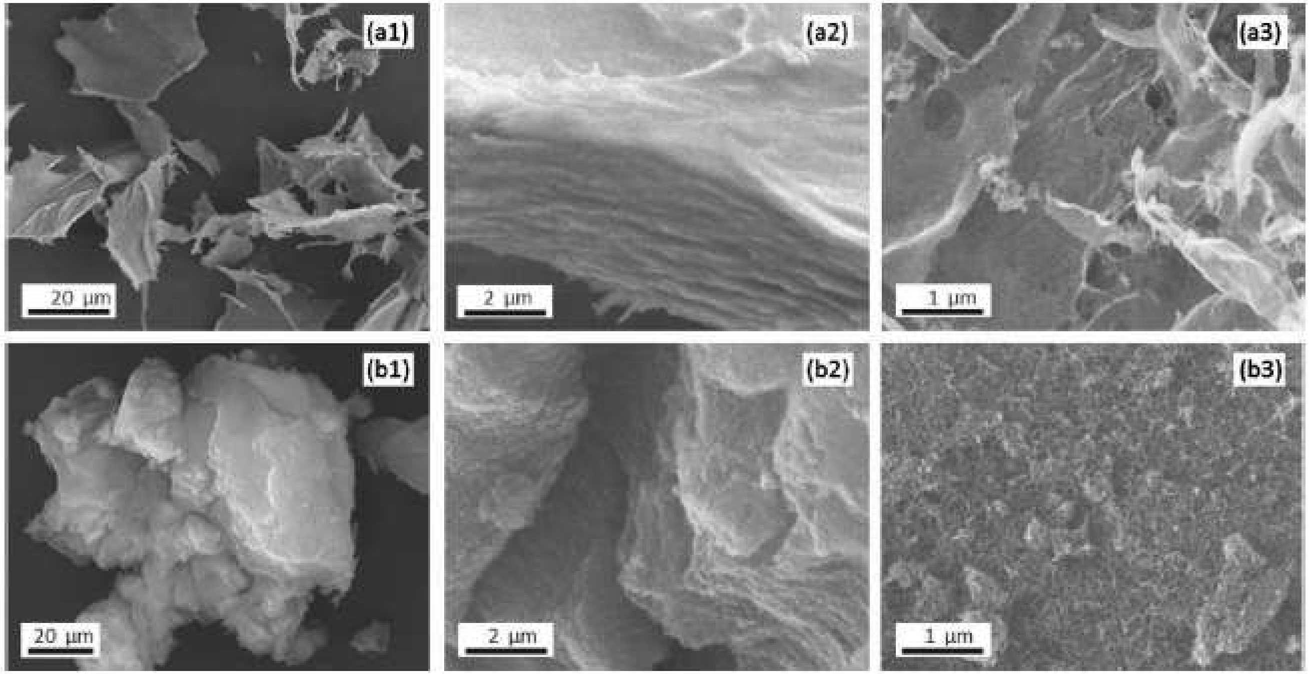


Fig. 1: SEM micrographs of lyophilized (a1–a3) and critical point dried (b1–b3) titania aerogels.

LYO:

- Well separated very thin foils (a2)
- Rather smooth surface (a3)

CPD:

- Rather bulk material, signs of lamellar structure (b2)
- Highly porous consisting of randomly oriented very small, probably planar, units (b3)

[DOI 10.1515/pac-2016-1031]

Titania aerogels

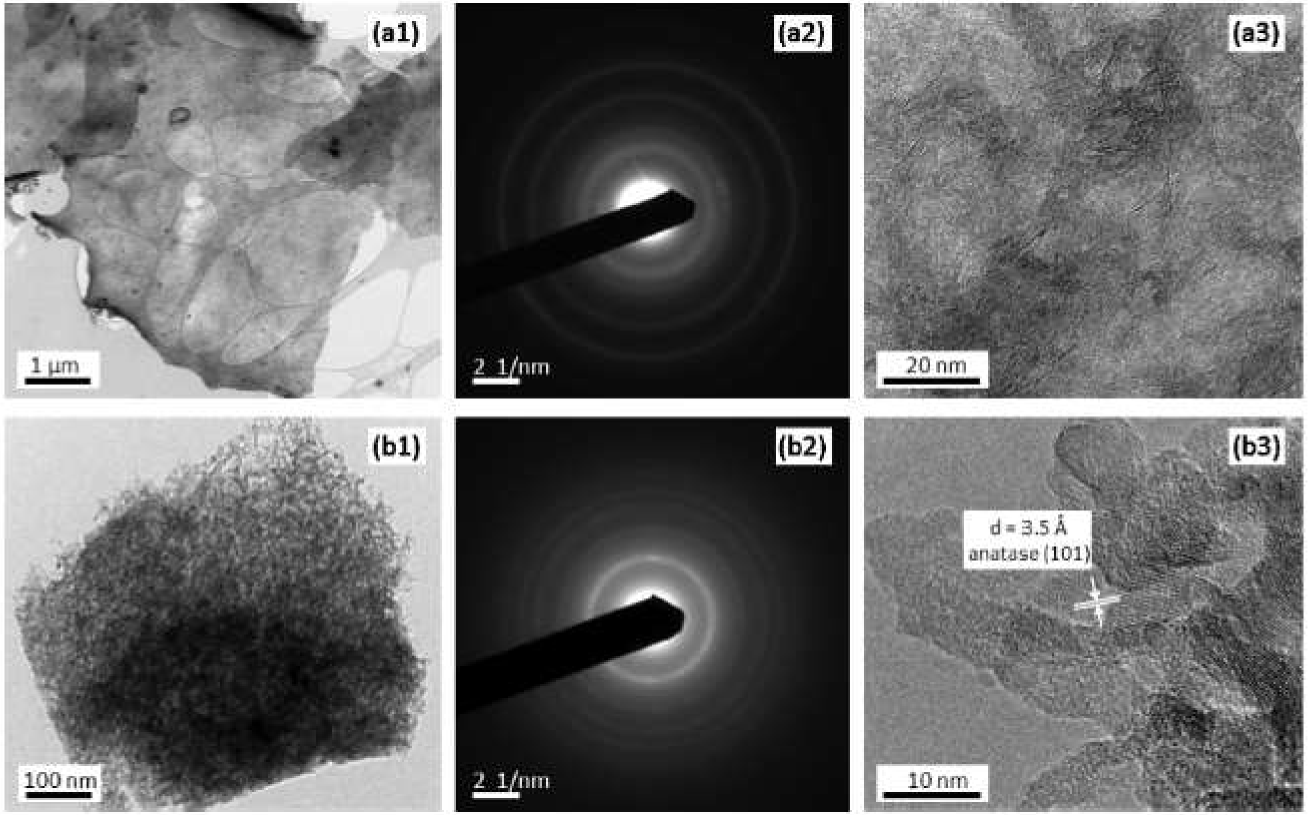


Fig. 2: TEM micrographs of lyophilized (a1–a3) and critical point dried (b1–b3) titania aerogels.

[DOI 10.1515/pac-2016-1031]

LYO:

- Foils have lateral size of several tens of µm, thickness of only about 10-20nm
- Composed of tightly packed grains
- SAED pattern: small grain size of 2nm bordering on amorphous character

CPD:

- 3D nano porous microstructure composed of flat nanoparticles about 5-10 nm an 2nm thickness
- Most composed of crystalline anatase

Titania aerogels

LYO:

- Suppose, that during fast freezing of nanoparticle containing aqueous colloids aggregate on the surface of ice grains
- Highly porous and voluminous aerogels consisting solely of the respective material can be obtained, by fast freezing aqueous colloidal nanoparticle suspension as droplets injected directly into liquid nitrogen

CPD:

- Not affected by the effect of growing ice grains
- Probably closer to the structure of the original aqueous colloid

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